

**NORTH ATLANTIC TREATY ORGANIZATION
ORGANISATION DU TRAITE DE L'ATLANTIQUE NORD**

**MILITARY AGENCY FOR STANDARDIZATION (MAS)
BUREAU MILITAIRE DE STANDARDISATION (BMS)
1110 BRUSSELS**

5 June 2001

MAS/0737-PPS/4147

See AC/310 STANAG distribution

**STANAG 4147 PPS (EDITION 2) – CHEMICAL COMPATIBILITY OF AMMUNITION
COMPONENTS WITH EXPLOSIVES (NON-NUCLEAR APPLICATIONS)**

References:

- a. AC/310-D/175 dated 14 December 1999
- b. MAS/170-MMS/4147 dated 1 June 1992 (Edition 1)

1. The enclosed NATO Standardization Agreement which has been ratified by nations as reflected in page (iii) is promulgated herewith.
2. The references listed above are to be destroyed in accordance with local document destruction procedures.
3. AAP-4 should be amended to reflect the latest status of the STANAG.

ACTION BY NATIONAL STAFFS

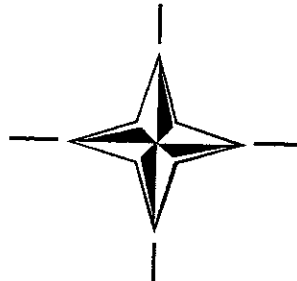
4. National staffs are requested to examine page (iii) of the STANAG and, if they have not already done so, advise the Defence Support Division through their national delegation as appropriate of their intention regarding its ratification and implementation.



Jan H ERIKSEN
Rear Admiral, NONA
Chairman, MAS

Enclosure:
STANAG 4147 (Edition 2)

**NORTH ATLANTIC TREATY ORGANIZATION
(NATO)**



**MILITARY AGENCY FOR STANDARDIZATION
(MAS)**

**STANDARDIZATION AGREEMENT
(STANAG)**

SUBJECT: CHEMICAL COMPATIBILITY OF AMMUNITION COMPONENTS
WITH EXPLOSIVES (NON-NUCLEAR APPLICATIONS)

Promulgated on 5 June 2001

A handwritten signature in black ink, appearing to read 'Jan H ERIKSEN'. The signature is fluid and cursive, with a long horizontal stroke at the end.

Jan H ERIKSEN
Rear Admiral, NONA
Chairman, MAS

STANAG 4147
(Edition 2)

RECORD OF AMENDMENTS

No.	Reference/date of amendment	Date entered	Signature

EXPLANATORY NOTES

AGREEMENT

1. This NATO Standardization Agreement (STANAG) is promulgated by the Chairman MAS under the authority vested in him by the NATO Military Committee.
2. No departure may be made from the agreement without consultation with the tasking authority. Nations may propose changes at any time to the tasking authority where they will be processed in the same manner as the original agreement.
3. Ratifying nations have agreed that national orders, manuals and instructions implementing this STANAG will include a reference to the STANAG number for purposes of identification.

DEFINITIONS

4. Ratification is "In NATO Standardization, the fulfilment by which a member nation formally accepts, with or without reservation, the content of a Standardization Agreement" (AAP-6).
5. Implementation is "In NATO Standardization, the fulfilment by a member nation of its obligations as specified in a Standardization Agreement" (AAP-6).
6. Reservation is "In NATO Standardization, the stated qualification by a member nation that describes the part of a Standardization Agreement that it will not implement or will implement only with limitations" (AAP-6).

RATIFICATION, IMPLEMENTATION AND RESERVATIONS

7. Page (iii) gives the details of ratification and implementation of this agreement. If no details are shown it signifies that the nation has not yet notified the tasking authority of its intentions. Page (iv) (and subsequent) gives details of reservations and proprietary rights that have been stated.

FEEDBACK

8. Any comments concerning this publication should be directed to NATO/MAS - Bvd Leopold III, 1110 Brussels - BE.

NAVY/ARMY/AIR

NATO STANDARDIZATION AGREEMENT
(STANAG)

CHEMICAL COMPATIBILITY OF AMMUNITION COMPONENTS WITH EXPLOSIVES
(NON-NUCLEAR APPLICATIONS)

Annexes:

- A - Scope of the Agreement
- B - Requirements and Assessment Criteria
- C - Principles Governing Compatibility Tests
- D - Test Procedures and Acceptance Limits

Related documents: none

AIM

1. The aim of this agreement is to standardize the means of assessment, including test procedures, by which the chemical compatibilities of explosives, pyrotechnics, primary explosives and propellants (referred to throughout this document as explosives) with other substances or other explosives used in armament manufacture are judged. The test procedures and criteria have been studied and agreed in order to facilitate cross-procurement and to provide a means whereby countries can be assured that ammunition and other explosives stores received from abroad have been designed and manufactured with proper regard to the need for chemical compatibility between explosives, or explosive and non-explosive components.

AGREEMENT

2. Participating nations agree that the scope of the agreement as described in Annex A, the Requirements and Assessment Criteria in Annex B, the Principles in Annex C and the Test Procedures and Acceptances Limits in Annex D shall form the basis upon which chemical compatibility of explosives with other ammunition components shall be judged.

WARNING

3. This STANAG calls for the use of substances and test procedures that may be injurious to health if adequate precautions are not taken. It refers only to technical suitability and in no way absolves the user from statutory obligations relating to health and safety at any stage during use.

IMPLEMENTATION

4. The STANAG is considered implemented by a nation when that nation has issued instructions that the test procedures for judging the chemical compatibility of ammunition components with explosives will be in accordance with the procedures detailed in this agreement.

SCOPE OF THE AGREEMENT

1. The STANAG is limited to matters relating to chemical compatibility of munition components with explosives and does not extend to procedures and requirements which concern the avoidance of incompatibility from other causes.

Attention is necessarily drawn to these causes which, for explosives, include:
 - (i) Migration of soluble or volatile ingredients at contact surfaces or through the vapour phase, leading to unacceptable changes in physical properties.
 - (ii) Changes in the sensitivity of the explosive to initiation by shock, friction or other external stimuli.
 - (iii) The introduction of an unacceptable risk of initiation by electrostatic discharge.
2. The STANAG is not concerned with compatibility between ingredients in explosive compositions and the consequent stability of such compositions.
3. The requirements of Annex B and the tests in Annex D ensure that the chemical compatibility of explosives with other ammunition components is at the necessary standard for safety during manufacture, storage and use, and for reliability after storage under approved conditions.
4. Explosives vary widely in their nature and properties and standardization of all compatibility test procedures is unlikely to be achieved. To assist in the harmonization of procedures to the maximum extent, Annex C presents principles to which acceptable compatibility tests should conform and which apply in cases where an agreed procedure from Annex D is not used.
5. The test Procedures in Annex D provide data which relate to the extent to which explosives react or decompose as a consequence of contact or proximity with the materials under test. The properties of explosives are diverse and no single test method is capable of overall applicability. A statement of applicability in each procedure provides guidance on the suitability of the procedure for the different classes of explosives.
6. A ratifying country which signifies its agreement to Procedures in Annex D will state in its acceptance the classes of explosives for which it accepts the Procedures as valid.

REQUIREMENTS AND ASSESSMENT CRITERIA

1. Conformity to Test Procedures and Acceptance Limits in Annex D is proof of chemical compatibility within the limits of applicability stated in the Test Procedures.
2. The results from one test alone, in particular the vacuum stability test when applied to propellants, should not necessarily be accepted as indicating compatibility and should whenever possible be supported by evidence from other tests based on a different principle.

If the result of the second test is "failed", the substance is not compatible with the explosive.
3. Test Procedures which are not included in Annex D must conform to the principles stated in Annex C. In such cases a special (or ad hoc) agreement on the procedure proposed by a selling country will be reached.
4. Confirmation of compatibility by testing may be waived by special (or ad hoc) agreement when the material complies with the requirements of paragraph 5 of this Annex and is shown to have had previous use in conjunction with the same or very similar explosives, which demonstrates its compatibility and suitability for a proposed application. Information from accelerated ageing trials may also be presented in support of statements of satisfactory compatibility.
5. To be accepted as compatible a material must conform to the requirements of the relevant specification. It must be subject to quality control procedures which ensure that its chemical composition corresponds to that of the specimen or supply whose behaviour in a compatibility test or in previous usage provides the basis of the acceptance. Alternatively, if the above requirements cannot be met, the testing of each lot or consignment of the material for compatibility is required.

PRINCIPLES GOVERNING COMPATIBILITY TESTS

1. GENERAL

- 1.1 The purpose of a compatibility test in the context of this STANAG is to provide evidence that a material may be used in an item of ammunition without detriment to the safety or reliability of an explosive with which it is in contact or proximity (see Annex A, paragraphs 1, 2 and 3).
- 1.2 The tests and acceptance criteria must provide for judgments of satisfactory compatibility with adequate margins of confidence but should, as far as is practicable, avoid the rejection of otherwise satisfactory material because of observed but unimportant levels of reactivity.
- 1.3 The methods of monitoring reactivity during heating tests or of examination subsequent to accelerated aging should preferably provide quantitative data. They must be appropriate to the explosive being tested and its required application.

This STANAG describes the following tests and procedures:

- vacuum stability (Test 1)
- heat flow calorimetry (Test 2)
- thermogravimetric analysis (Test 3)
- differential scanning calorimetry (Test 4)
- chemical analysis after ageing (Test 5)

One or more of the above tests as appropriate may be used to determine the compatibility of the test material with explosives.

Other test methods may also be used if it can be demonstrated that it is more appropriate to the test material and explosives being examined.

However if the procedures used can only provide qualitative information and any adverse reaction is detected, then the material under test should be regarded as incompatible. Information should also be provided on the level at which reactivity leading to incompatibility becomes detectable.

- 1.4 Valid test procedures require that a combination of explosive and test material be heated together at a temperature and for a time which can reasonably be considered to be equivalent to the anticipated storage and service life. The stringency of the procedure is dependent upon the temperature and time of heating but the quantitative effects of temperature upon the rates of reactions between particular explosives and test materials will not as a rule be known or investigated unless the compatibility problem is of special importance.

The minimum duration versus temperature is given with each test.

ANNEX C to
STANAG 4147
(Edition 2)

- 1.5 The proportions of test material to explosive and the degree of contact which is achieved by mixing them, have a marked effect upon the extent of reaction which is observed in compability tests. The normal proportion for mixing is 1: 1, with the exception of test 5.

However variations of the proportions in either direction may be appropriate in some circumstances, depending upon the nature of the tests, of the test materials and explosives, and of the design features affecting the juxtaposition of the explosive and material in the ammunition for which the test is being conducted. For example, when the explosive is to be used in very small amounts, it may be necessary to test for the effects which much larger amounts of the test material may have upon it. The converse situation may also arise.

- 1.6 The influence of moisture on incompatibility reactions is frequently important, as is the degree of confinement of the test mixture and the availability of atmospheric oxygen and the presence of solvent during the accelerated aging heating period. Testing authorities should define the conditions controlling the above variables during their tests and be prepared to present an explanation for them.
- 1.7 At the end of or during the heating period the reactivity of the test material with the explosive may be measured with the object of determining the extent to which the explosive has reacted or decomposed. The procedure must include parallel testing of the explosive alone and separate testing of the test material so that the reactivity due to incompatibility may be distinguished from any intrinsic instability of the separate explosive or test material under the test condition.
- 1.8 The choice of acceptance criteria for procedures which provide quantitative data may be made in two alternative ways:
- a. relative criteria: by reference to the extent of the decomposition suffered by the explosive in the test in the absence of incompatibility (A proportionate increase in decomposition will be allowed depending upon the severity of the test and the stability properties of the explosive. In general, the criteria for compatibility with less stable explosives will be more stringent).
 - b. absolute criteria: arbitrarily being set at a level which will restrict the observed reactivity to a safe acceptable level.

Alternative (a) is usually preferable to (b), the latter being necessary for explosives which do not normally decompose to a measurable extent under the test condition. Testing authorities using an unstandardized test should be prepared to explain the basis for their acceptance criterion.

- 1.9 Many explosive compositions differ to only a minor extent from other related compositions. Compatibility data applicable to one such explosive may give an indication of the compatibility of related compositions. It is not possible to define limits within which such judgments may be admissible but their acceptability when agreed between the parties concerned is to be recognised as consistent with the requirements of this STANAG.
- 1.10 Some materials to be tested for compatibility may contain solvents or impurities which could vary from one lot to another and affect the results obtained. Consequently it is important to uniquely identify the material by lot number and commercial reference.

2. EXPLOSIVES PROPERTIES AND TESTS

In this section the compatibility properties of the various groups of explosives are considered and the principles of suitable tests are indicated. The grouping of the explosives is based upon their chemical properties and in particular upon their more frequent modes of decomposition or incompatibility.

2.1 High Explosives

Explosives containing the following and chemically related substances as principal ingredients are included in this group: TNT, RDX, HMX, tetryl, PETN, HNS, TATB, ammonium nitrate. Explosives containing nitroglycerine or other liquid nitrate esters, as one of the principal ingredient, are not in this category for compatibility consideration. They may fall more appropriately into Section 2.2.

Incompatibility with high explosives can be determined using tests measuring gas evolution, weight loss, and also measuring heat change. Tests 1, 2, 3 and 4, in Annex D are applicable in determining incompatibility with high explosives.

Materials in contact with these explosives require to be free from strong acidity or alkalinity.

2.2 Propellants containing Nitrocellulose as a Principal Ingredient

These comprise single, double and triple base propellants usually manufactured by extrusion or casting. An increased rate of decomposition caused by incompatibilities with these propellants can be determined using tests measuring, gas evolution, heat generation, weight loss, decomposition kinetics or loss of stabilizer content. Tests 2, 3, 4 and 5, procedure A, in Annex D are applicable in determining incompatibility with these propellants. Test 1 is considered applicable by several countries but may not be accepted by all countries unless supported by data from other tests.

Although properly formulated propellants of this class, prepared from stable ingredients, can be expected to remain safe in storage at normal temperatures over many years, they contain ingredients of comparatively low stability and standards for compatibility acceptance are required to be comparatively severe.

Test methods should provide quantitative data for the rates of decomposition of the propellant in the presence and absence of the test material and so that the comparative effect of any reactivity on the stability of the propellant and its safe life can be judged.

Materials in contact with this type of propellant require to be free from strong acidity or alkalinity.

ANNEX C to
STANAG 4147
(Edition 2)

2.3 Pyrotechnic Explosives

Numerous explosives with widely differing compositions fall into this category and exceptions to the following generalised comments may arise. Most pyrotechnic explosives are intimate mixtures of substances which are themselves individually stable but capable of reacting together to produce the desired effect. Problems of instability or incompatibility with such mixtures are frequently increased significantly by the presence of moisture and test procedures should where necessary be designed to show that the material under test does not increase the susceptibility of the explosive to deterioration caused by moisture. Unless reasons are given for excluding moisture, the procedures should introduce an appropriate level of humidity so that the explosive alone is affected to a small but measurable extent. This will not apply to explosives containing ingredients which react readily with water and require protection from it.

Many pyrotechnic explosives are formulated to decompose or react without generating significant volumes of gas. Gas evolution tests are not generally applicable and methods based on thermal, chemical or physico-chemical analysis after periods of heating (in accordance with the principles described in Section 1 of this Annex) are preferred. Tests 3 and 4 in Annex D are considered suitable for determining incompatibility with pyrotechnic explosives.

2.4 Primary Explosives

Several of the above statements pertaining to pyrotechnic explosives are applicable to primary explosives. The numerous compositions may be either single substances or mixtures and many of the individual compounds which are used are thermally stable to fairly high temperatures. They produce only small volumes of gas when heated at desirable temperatures for compatibility tests, unless undesirably large quantities are used. Such tests, based on gas evolution measurement, are of doubtful value and methods based on thermal, chemical or physico-chemical analysis after periods of heating (Section 1) are preferred. Tests 4 and 5, procedure B in Annex D are considered suitable for determining incompatibility with primary explosives.

The stability and reactivity of several important primary explosives is much influenced by the presence of moisture and it will usually be advantageous to introduce an appropriate level of humidity into the conditions of testing. This is particularly important when testing lead azide.

Characteristically the use of primary explosives in ammunition requires only small charges to be present. These will exist in an assembly with much greater quantities of surrounding materials. If the compatibility of the explosive with these materials is in question, the tests must incorporate this disparity in amounts.

2.5 Composite Propellants

These propellants are generally composed of energetic materials bound together using a binder. These propellants tend to be thermally stable, but can suffer deterioration by exposure to moisture. Another cause of deterioration results, for example, from changes in the cross linking of the inert binder, leading to unacceptable changes in the mechanical properties of the propellant. Changes of this nature are not covered by this STANAG but tests used for monitoring such changes in mechanical properties can be found in STANAG's 4443, 4506, 4507, 4525 and other procedures described in Annex 1, Volume 2 of AOP-7. Tests 1, 2, 3 and 4 of this STANAG are considered suitable for determining any incompatibility with composite propellants, resulting in a deterioration in the thermal or chemical stability of the propellant.

TEST PROCEDURES AND ACCEPTANCE LIMITS

Test 1.	<u>Procedure A - The Vacuum Stability Test (Manometer Method)</u>	D-2
	APPENDIX 1 - TEST 1A - DATA SHEET.....	D-9
	APPENDIX 2 - TEST 1A - ACCEPTANCE LIMIT FOR TEST 1A.....	D-11
	<u>Procedure B - The Vacuum Stability Test (Transducer Method)</u>	D-13
	APPENDIX 1 - TEST 1B - DATA SHEET.....	D-20
	APPENDIX 2 - TEST 1B - ACCEPTANCE LIMIT FOR TEST 1B.....	D-22
	APPENDIX 3 - TEST 1B - FIGURE 1B.1.....	D-24
	APPENDIX 4 - TEST 1B - FIGURE 1B.2.....	D-25
Test 2	The Heat Flow Calorimetry Test.....	D-25
	TEST 2 - DATA SHEET.....	D-29
TEST 3	<u>PROCEDURE A - DYNAMIC THERMOGRAVIMETRY (TGA)</u>	D-31
	<u>PROCEDURE B - ISOTHERMAL THERMOGRAVIMETRY (TGA)</u>	D-36
	APPENDIX 1 - TEST 3 B - FIG.3B.1.....	D-39
	APPENDIX 2 - TESTS 3 A & B - DATA SHEET	D-39
	<u>PROCEDURE C - DETERMINATION OF THE KINETICS OF DECOMPOSITION</u>	D-41
	APPENDIX 1 - TEST 3C - DATA SHEET TEST 3C DATA SHEET.....	D-45
TEST 4	DIFFERENTIAL SCANNING CALORIMETRY.....	D-47
	APPENDIX 1 - TEST 4 - FIG.4.1	D-51
	APPENDIX 2 TEST 4 - DATA HSEET.....	D-51
TEST 5	CHEMICAL ANALYSIS.....	D-53
	<u>PROCEDURE A - CHEMICAL ANALYSIS - ASSESSMENT OF THE COMPATIBILITY OF</u> <u>AMMUNITION COMPONENT MATERIALS WITH NITRATE D-ESTER BASED PROPELLANTS</u>	D-54
	APPENDIX 1 - TEST 5A - DATA SHEET.....	D-58
	APPENDIX 2 - TEST 5A - ACCEPTANCE LIMITS FOR PROCEDURE A.....	D-59
	APPENDIX 3 - TEST 5A - TEST 5A - FIGURE 5A.1	D-60
	<u>PROCEDURE B - CHEMICAL ANALYSIS - ASSESSMENT OF THE COMPATIBILITY OF</u> <u>AMMUNITION COMPONENT MATERIALS WITH LEAD AND SILVER AZIDE</u>	D-61
	APPENDIX 1 - TEST 5B - DATA SHEET	D-67
	APPENDIX 2 - TEST 5B - ACCEPTANCE LIMITS FOR PROCEDURE B.....	D-68
	APPENDIX 3 - TEST 5B - FIGURE 5B.1.....	D-70
	APPENDIX 4 - TEST 5B - SOME GUIDANCE NOTES ON THE USE OF AN AUTOMATIC TITRATOR FOR THIS PROCEDURE.....	D-70

TEST 1: PROCEDURE A

THE VACUUM STABILITY TEST (MANOMETER METHOD)

1. SUMMARY DESCRIPTION

The volume of gas evolved, when a mixture of equal parts of an explosive and the material under test is heated at a constant temperature of 100°C for 40 hours in an initial vacuum, is compared with the volumes evolved from the explosive and the test material when heated separately under otherwise identical conditions. Compatibility is judged by means of the volume of additional gas produced because of the contact between the two components of the mixture.

Most tests by this method are conducted at 100°C for 40 hours and this time and temperature can therefore be regarded as adequate. Tests at 80°C for 240 hours are considered adequate for double base propellants and explosives which decompose too rapidly when heated at 100°C. Temperatures much higher than 100°C are undesirable because the test conditions become increasingly unrealistic. Conversely, lower test temperatures are desirable and are essential for some types of test materials and explosives. Longer periods of test then become necessary. For tests conducted at below 100°C, the minimum times are multiplied by a coefficient equal to 2,5 for every temperature decrease of 10°C, for example 24 days at 70°C, 60 days at 60°C.

2. APPLICABILITY

The test is applicable to solid high explosives and propellants used in conventional armaments. However some authorities require supporting data from other tests when this test is applied to propellants.

3. INTERPRETATION OF RESULTS

Materials which fail to satisfy the requirements of this test will be judged as unsuitable for use in applications where permanent direct contact or close proximity with the test explosive or propellant is required or where adequate precautions to guard against such contact are not taken.

4. APPARATUS

4.1 Description of equipment

4.1.1 Constant temperature bath able to maintain the tube containing the sample at the desired temperature to within $\pm 0.5^\circ\text{C}$.

The following alternatives may be used:

- a. Electrically heated metal block controllable to within $\pm 0.2^\circ\text{C}$. The block shall contain holes provided with appropriate reducing sleeves and able to accommodate the total length of the sample heating tubes. The dimensions of the sleeves shall be such that the diameter is approximately 1 to 2 mm greater than the diameter of the heating tube.
- b. Thermostatically controlled bath filled with a liquid appropriate for the specified temperature controllable to within $\pm 0.2^\circ\text{C}$.

For each alternative an electrical cut-out shall be fitted which operates at 5°C above the nominal working temperature.

- 4.1.2 Vacuum stability measuring apparatus in glass comprising a heating tube and a capillary tube ending in a cup shaped reservoir (see Appendix n° 3).
- 4.1.3 Primary vacuum pump able to attain a pressure of less than 5 mm mercury.
- 4.1.4 Mercury thermometers or thermometric gauges calibrated 0 to 160°C, in 0.2°C.
- 4.1.5 Barometer.
- 4.1.6 Cathetometer controllable to within ± 1 mm.
- 4.1.7 Precision balance readable to 1 mg.
- 4.1.8 Distilled mercury, clean and dry.
- 4.1.9 Lubricant compatible: petroleum jelly or high vacuum silicone grease which do not evolve gas at the specified temperature for the test.
- 4.2 Calibration of the vacuum stability apparatus

Calibrate the vacuum stability measuring apparatus as follows.

4.2.1 Heating tube

Determine the volume of the heating tube to 0.1 cm³ by filling it with mercury (or other suitable liquid) from a burette until the liquid reaches the level at which it will contact the ground glass joint of the capillary tube.

$$v = \frac{m}{d}$$

v = volume, cm³

m = mass of liquid, g

d = density of liquid at the calibration temperature t°C, g.cm⁻³

ANNEX D to
STANAG 4147
(Edition 2)

4.2.2 Capillary tube

Determine the unit capacity of the capillary by placing approximately 10 g, weighed to the nearest 0.01 g, of mercury in the reservoir, and manipulate the tube so that all the mercury passes into the long (850 mm) section of the capillary. Ensure that the mercury remains as a continuous column. Measure the length at three positions in the long section of the capillary, and average the three measurements. Calculate the unit capacity of the capillary, using the following formula:

$$C = \frac{m}{L \times d}$$

where:

- C = unit capacity of capillary, $\text{cm}^3 \cdot \text{mm}^{-1}$.
 m = mass of mercury, g
 L = average length of mercury column, mm.
 d = density of mercury at the calibration temperature $t^\circ\text{C}$, $\text{g} \cdot \text{cm}^{-3}$

5. PREPARATION OF EXPLOSIVES

With necessary precautions, grind or subdivide the explosive to pass through a sieve with a 2 mm opening. Discard material which passes a 0.2 mm sieve. When the explosive contains a major ingredient (such as trinitrotoluene) which melts during the course of the test, a larger grist size is permissible. Explosives which do not require grinding or sub-division to pass a 2 mm sieve are not to be sieved before use to remove fine particles. The specimen shall be dried or prepared in accordance with the requirements for the explosive under test.

6. PREPARATION OF TEST MATERIALS

The material used for the test shall be, as far as is practical, in its proposed condition for contact with the explosive. Where the intended use requires the test material to be applied in an intermediate (uncured or partly cured) condition to the explosive, the compatibility test shall be made on mixtures so prepared and then treated in the manner prescribed to bring the test material to its final (cured) condition.

Most solid materials require to be subdivided for testing to obtain effective mixing and a high degree of contact with the explosive during the test.

Subdivision of solid materials in block form must reduce them to particles with a maximum size of 2 mm and the preparation of materials as shavings or by cutting up of thin sheets or other means must allow a degree of mixing and contact with the explosive which is at least as effective. Discard material that has been produced by the process of subdivision and which passes a 0.2 mm sieve.

Test materials should not be artificially dried before testing but the presence of excessive quantities of volatile ingredients may make the test impossible to perform. To remove excessive quantities of water, preliminary drying over silica gel for 24 hours at ambient temperature is permitted. To remove volatile solvents other methods may be used. These treatments must be recorded in the test report.

7. PRELIMINARY TEST

When testing materials of unknown chemical reactivity it is desirable to confirm by means of simple tests, in which small quantities are mixed with explosives and warmed, that reactions between the substances being tested for compatibility are not liable to cause explosion or fire during the test.

8. COMPATIBILITY TEST

- 8.1 Mix 2.5 g. of the test explosive with 2.5 g. of the test material and transfer the mixture to the heating tube of the apparatus, taking care to avoid contamination of the ground glass joint of the tube (the introduction of the explosive can be facilitated by the use of a wide-bore short-stemmed glass funnel). A minimum of two tests shall be conducted.
- 8.2 Coat the ground glass joint of the capillary tube with a light film of the lubricant (use the minimum amount of lubricant to ensure a good joint). Make an airtight connection between the heating tube and the capillary by pressing the tube up against the capillary with a gentle twisting motion, until the interface of the joint is clear.
- Take care to prevent the tube falling from the manometer before it is held by the vacuum.
- 8.3 Mount the assembled apparatus in a suitable rack so that the long section of the capillary is nearly vertical and the bottom reservoir rests on a solid support.
- 8.4 Add to the capillary reservoir a sufficient amount of clean mercury to fill the capillary and the reservoir after evacuation (about 7 cm³ or one-third filled).
- 8.5 Connect the capillary reservoir to the vacuum pump using a vacuum line and rubber adapter. Tilt the assembly to bring the reservoir towards the horizontal until the capillary opening is free of mercury. Evacuate the heating tube and manometer until the pressure is reduced to a maximum of 5 mm of mercury. Tap the heating tube lightly to facilitate release of any occluded air from the sample.
- 8.6 When the evacuation is completed, return the capillary to the vertical. Allow mercury to enter the capillary by slowly admitting air into the vacuum line through the stopcock. This operation must be done very carefully to prevent the mercury rising too quickly in the manometer tube. Disconnect the vacuum line. Pour a little mercury into the cup of the heating tube to serve as a secondary seal and cover the mercury with a thin layer of glycerol or water to prevent evaporation of the mercury.

ANNEX D to
STANAG 4147
(Edition 2)

8.7 Record the following data

- The total length of the capillary tube minus the vertical height of the column of mercury in the reservoir before heating (B_1) to 1 mm.
- The height of the mercury column above the surface of the mercury in the reservoir at the beginning of the test (H_1) to 1 mm.
- The room temperature at the beginning of the test (t_1) to 0.5°C.
- The barometric pressure at the beginning of the test (P_1) to 0.5 mm Hg.

8.8 Ascertain that the bath temperature is constant at the specified temperature. Carefully place the heating tube of the prepared test assembly in the constant temperature bath. The whole heating tube (with its ground glass joint) must be placed in the constant temperature bath, taking care not to loosen the connection between the heating tube and the capillary. Support the capillary reservoir and heat the tube for the specified heating period.

8.9 Remove the tube from the constant temperature bath and allow to cool to room temperature.

8.10 Record the following data

- The total length of the capillary tube minus the vertical height of the column of mercury in the reservoir after heating (B_2) to 1 mm.
- The height of the mercury column above the surface of the mercury in the reservoir at the end of the test (H_2) to 1mm.
- The room temperature at the end of the test (t_2) to 0.5°C.
- The barometric pressure at the end of the test (P_2) to 0.5 mm Hg.

8.11 Execute the same tests with

- An heating tube containing only 2.5 g of explosive.
- An heating tube containing only 2.5 g of the test material.

Heat both samples, using identical test conditions as those used for heating the mixture.

9. CALCULATION

9.1 Calculate the volume of gas V at standard temperature and pressure liberated from the sample during the test, as follows:

$$V = [A + C(B_2 - H_2)] \times \frac{273(P_2 - H_2)}{760(273 + t_2)} - [A + C(B_1 - H_1)] \times \frac{273(P_1 - H_1)}{760(273 + t_1)}$$

Where:

- V = volume of gas liberated from the sample (cm^3 , at STP).
 A = volume of the tube minus the volume of the sample (cm^3).
 B_1 = total length of the capillary tube minus the vertical height of the column of mercury in the reservoir at the beginning of the test (mm).
 B_2 = total length of the capillary tube minus the vertical height of the column of mercury in the reservoir at the end of the test (mm).
 C = unit capacity of the capillary tubing ($\text{cm}^3 \cdot \text{mm}^{-1}$).
 H_1 = height of the mercury column above the surface of the mercury in the reservoir at the beginning of the test (mm).
 H_2 = height of the mercury column above the surface of the mercury in the reservoir at the end of the test (mm).
 P_1 = barometric pressure at the beginning of the test (mm Hg).
 P_2 = barometric pressure at the end of the test (mm Hg).
 t_1 = room temperature at the beginning of the test ($^{\circ}\text{C}$).
 t_2 = room temperature at the end of the test ($^{\circ}\text{C}$).

- 9.2 Calculate with the formula above, the volumes of gas evolved by the mixture and by the explosive and the test material when heated separately.

- 9.3 Calculate:

$$V_R = M - (E + S)$$

Where:

- V_R = Volume of gas produced as a consequence of reaction between the components of the test mixture.
 M = Volume of gas from 2.5 g of explosive mixed with 2.5 g of test material (cm^3 at STP)
 E = Volume of gas from 2.5 g of explosive (cm^3 , at STP)
 S = Volume of gas from 2.5 g of test material (cm^3 , at STP)

- 9.4 Report the results of the test on the Data Sheet (Appendix 1 to Test 1 Procedure A).

ANNEX D to
STANAG 4147
(Edition 2)

10. DISMANTLING

Remove the mercury from the cup of the heating tube by suction and connect the capillary reservoir to the vacuum pump. Tilt the assembly and evacuate the capillary and heating tube. Admit air to the apparatus taking care that the heating tube does not fall from the capillary. Return the capillary to the vertical position and disconnect it from the vacuum pump. Pour out the mercury into a bottle kept specially for "dirty" mercury and separate the capillary and heating tube for cleaning.

11. CLEANING

Empty the heating tube and remove residual traces of the sample and any lubricant round the joint with suitable solvents. Rinse with acetone and then with water. Fill the heating tube and capillary with a suitable solution for glassware cleaning and allow to stand for 24 hours. Finally rinse with water followed by acetone and blow dry with clean, dry air. Store the cleaned heating tubes in a desiccator until required for use.

TEST 1A - DATA SHEET

NATO/PfP UNCLASSIFIED

NATO STANAG 4147 DATA SHEET (Side B)	
Report Reference Number: (Unique Reference Number)	Page of Page(s)
DIVERGENCES FROM STANDARD PROCEDURE: (if applicable include details of drying, curing or solvent removal)	
TEST RESULTS: (Record the values of M, E, S and $V_R \text{ cm}^3$ at STP) Volume of gas from 5 g of admixture (M): Volume of gas from 2.5 g of explosive (E): Volume of gas from 2.5 g of test material (S): $V_R = M - (E + S)$	
COMPATIBLE: YES NO	
DATA SENT TO: (Name and address of person receiving this information)	
COMMENTS:	

TEST 1A

ACCEPTANCE LIMIT FOR TEST 1A

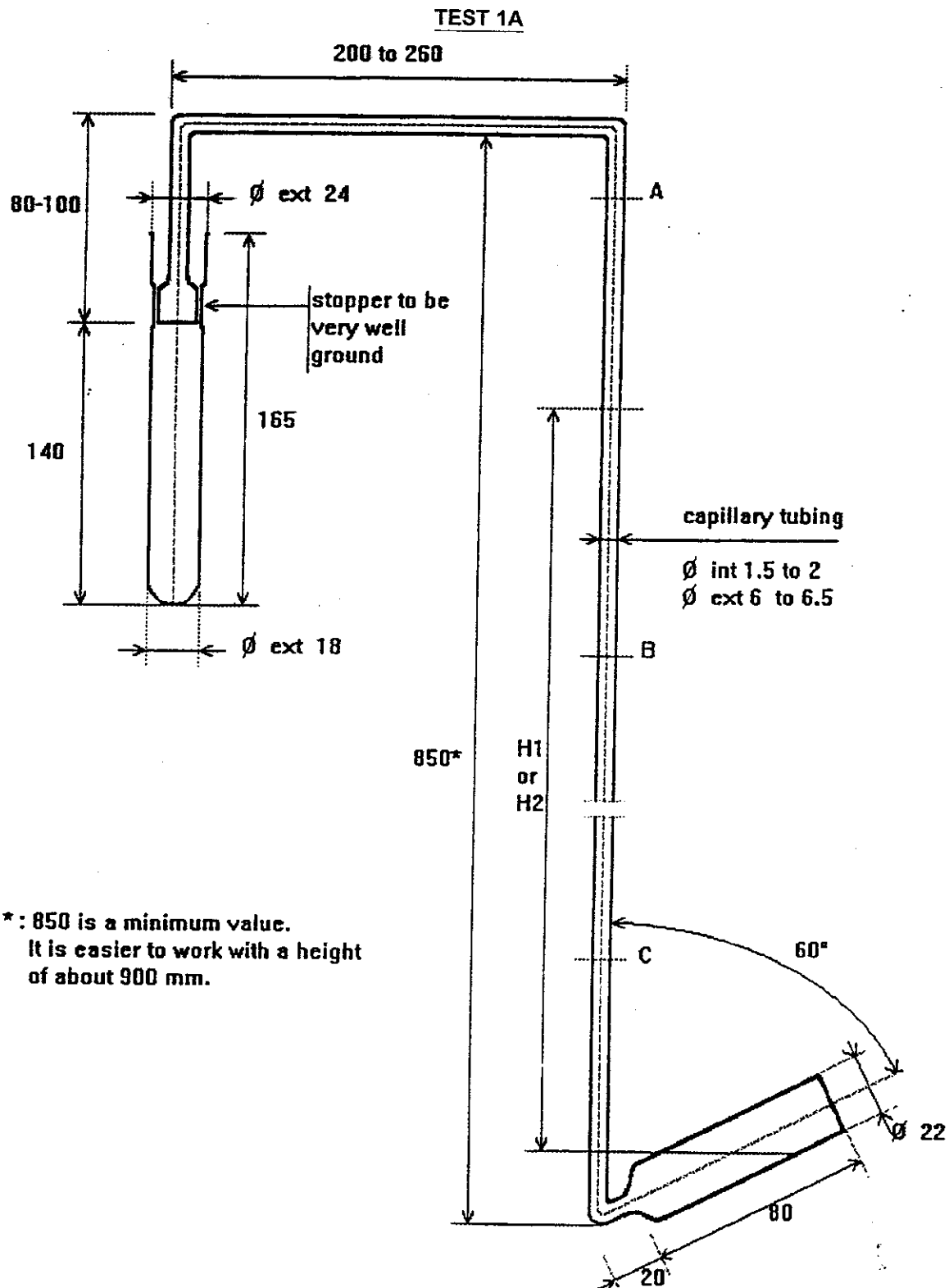
1. For materials to which paragraph 2 of this Appendix does not apply, the volume of gas $V_R = M - (E + S)$ must not exceed 5 cm^3 at STP where:

M = volume of gas evolved by 2.5 g of the test explosive when mixed with 2.5 g of the test material and heated for the specified time under the required test condition.

E = volume of gas evolved by 2.5 g of the test explosive heated for the specified time under the required test condition.

S = volume of gas evolved by 2.5 g of the test material heated for the specified time under the required test condition.
2. Materials supplied to specifications which include requirements for testing for compatibility with high explosives or propellants by the Vacuum Stability Test, and for which the permitted volume of evolved gas V_R , as defined in paragraph 1 of this Appendix, is less than 5 cm^3 at STP must be tested and sentenced in accordance with the requirements of their specifications.
3. When V_R approaches 5 cm^3 , retest is recommended to confirm the result. Confirmation, using another test based on a different principle, should also be sought. If the second test is "failed", then the test material is not compatible with the explosive.

Appendix 3 to
TEST 1A of
ANNEX D to
STANAG 4147
(Edition 2)



TEST 1: PROCEDURE B**THE VACUUM STABILITY TEST (TRANSDUCER METHOD)****1. SUMMARY DESCRIPTION**

The volume of gas evolved, when a mixture of equal parts of an explosive and the material under test is heated at a constant temperature of 100°C for 40 hours in an initial vacuum, is compared with the volumes evolved from the explosive and the test material when heated separately under otherwise identical conditions. Compatibility is judged by means of the volume of additional gas produced because of the contact between the two components of the mixture.

Most tests by this method are conducted at 100°C for 40 hours and this time and temperature can therefore be regarded as adequate. Tests at 80°C for 240 hours are considered adequate for double base propellants and explosives which decompose too rapidly when heated at 100°C.

Temperatures much higher than 100°C are undesirable because the test conditions become increasingly unrealistic. Conversely, lower test temperatures are desirable and are essential for some types of test materials and explosives. Longer periods of test then become necessary.

For tests conducted at below 100°C, the minimum times are multiplied by a coefficient equal to 2.5 for every temperature decrease of 10°C, for example 24 days at 70°C, 60 days at 60°C.

2. APPLICABILITY

The test is applicable to solid high explosives and propellants used in conventional armaments. However some authorities require supporting data from other tests when this test is applied to propellants.

3. INTERPRETATION OF RESULTS

Materials which fail to satisfy the requirements of this test will be judged as unsuitable for use in applications where permanent direct contact or close proximity with the test explosive or propellant is required or where adequate precautions to guard against such contact are not taken.

4. APPARATUS**4.1 Description of equipment.****4.1.1 Constant temperature bath-metal block type.**

Electrically heated metal block controllable to within $\pm 0.2^\circ\text{C}$. The block shall contain holes provided with appropriate reducing sleeves and able to accommodate the total length of the sample heating tubes + transducers (if possible). The dimensions of the sleeves shall be such that the diameter is approximately 1 to 2 mm greater than the diameter of the heating tube.

ANNEX D to
STANAG 4147
(Edition 2)

4.1.2 Transducers (See Appendix 3 and 4)

The transducers must be capable of operation in the pressure range 0 to 760 mm Hg (0 to 1 bar) absolute and be able to detect a variation in volume of $\pm 0.02 \text{ cm}^3$. The transducers should be allowed to stabilize for approximately 30 minutes after power up.

These transducers convert a fluid's variation to pressure exerted on a membrane into an electric signal by the imbalance of a Wheatstone bridge formed by four active strain gauges.

Each transducer shall be accompanied by its own calibration certificate supplying the information necessary for the conversion of the voltage measured into pressure. This formula is of a polynomial type. For method N°1 corrections must be applicable to transducers not temperature-compensated when they are used at a temperature different to that at which they were calibrated.

The transducers can be attached to the heating tubes as follows:

- a. with the steel male part of a ground metal joint. This part is welded to the transducer and includes a hole for gas extraction (see Appendix 5);
- b. with an adapter which attaches to the transducer using a hexagon nut connection. A gas tight fit is made to the heating tube using two "O" rings. The adapter includes an evacuation port. (see Appendix 4).

4.1.3 Glass heating tubes (See Appendix 3 and 4)

These tubes should be of uniform bore, with a wall thickness of 2 mm. All the tubes must be fitted to the pressure transducers so that each transducer + tube assembly has a free volume of $25 \pm 2 \text{ cm}^3$.

Examples of suitable test apparatus are shown in Appendix 3 and 4.

Other suitable glass heating tube/transducer assemblies may be used providing a free volume of $25 \pm 2 \text{ cm}^3$ is maintained. The type of assembly used and any changes to the calibration procedure required to accommodate the alternative apparatus shall be recorded in the data sheet.

- 4.1.4 Digital voltmeters, or data-logging equipment, capable of measuring the output voltage of the pressure transducer used.
- 4.1.5 Primary vacuum pump able to attain a pressure of less than 5 mm mercury.
- 4.1.6 Mercury thermometers or thermometric gauges calibrated, 0 to 160°C, in 0.2°C.
- 4.1.7 Barometer.
- 4.1.8 Pressure gauge indicating the range 0 to 20 mm absolute.
- 4.1.9 Precision balance readable to 1 mg.
- 4.1.10 Lubricant compatible: petroleum jelly or high vacuum silicone grease which does not evolve gas at the specified temperature for the test.
- 4.1.11 Calibrated gas tight syringe of 5 cm³ capacity.

4.2 Measuring of the free volumes.4.2.1 Method N° 1 (for apparatus shown in Appendix 3)

a. Heating tube.

Determine to the nearest 0.1 cm³ the volume of the test tube in the apparatus by filling it with a standard liquid up to the point at which the tapered part of the transducer stops when it is fitted to the tube.

Let:

m be the mass in g of the standard liquid used

ρ be the density of the standard liquid in g.cm⁻³ used at the measurement temperature.

V_t be the tube volume.

$$V_t = \frac{m}{\rho}$$

b. Transducer gauge.

With the transducer's gas evacuation hole plugged (by means of adhesive tape, for example), the transducer is filled with standard liquid up to the end of its tapered part.

Let:

V_c be the transducer volume

$$V_c = \frac{m}{\rho}$$

4.2.2 Method N° 2 (for apparatus shown in Appendix 4)

For this method the test material, explosive and admixture must be in the heating tube during calibration. See Section 5 , 6 and 8.1 for the required procedures.

Transfer the specified mass of the prepared specimen to the heating tube (see Section 8.1). Care shall be taken to ensure that the neck of the tube is free from adhering particles. (The introduction of the specimen can be facilitated by the use of a wide-bore short-stemmed glass funnel).

Lightly smear the "O" rings of the adapter with the lubricant. Assemble the apparatus as in Appendix 4. Connect the adapter evacuation port to the vacuum line, slide the inner tube outwards and evacuate the tube to less than 5 mm. When evacuation is complete, indicated by a steady reading of the digital voltmeter, seal the evacuation port by sliding the inner adapter tube inwards. Check that the seal is effective by monitoring for any drift on the voltmeter. Disconnect the vacuum line and record the voltmeter reading R_0 . Set the plunger of the calibrated syringe to the required calibrating volume (3 cm³) and connect to the adapter port. Inject 3.0 cm³ of air into the heating tube. Record the voltmeter reading R_3 .

Inject a further 5.0 cm³ of air and record the digital voltmeter reading R_5 , the barometric pressure, P mm, and the room temperature t°C.

ANNEX D to
STANAG 4147
(Edition 2)

The sample vessel is closed gastight to prevent volatile products from escaping. The vessel can be closed by means of an inert membrane. The sample inside the vessel will be in an air atmosphere.

The sensitivity of the apparatus should be $5 \mu\text{W.g}^{-1}$ or better. The precision should be at least 30% at $5 \mu\text{W.g}^{-1}$ and 5% from 100 to $1500 \mu\text{W.g}^{-1}$.

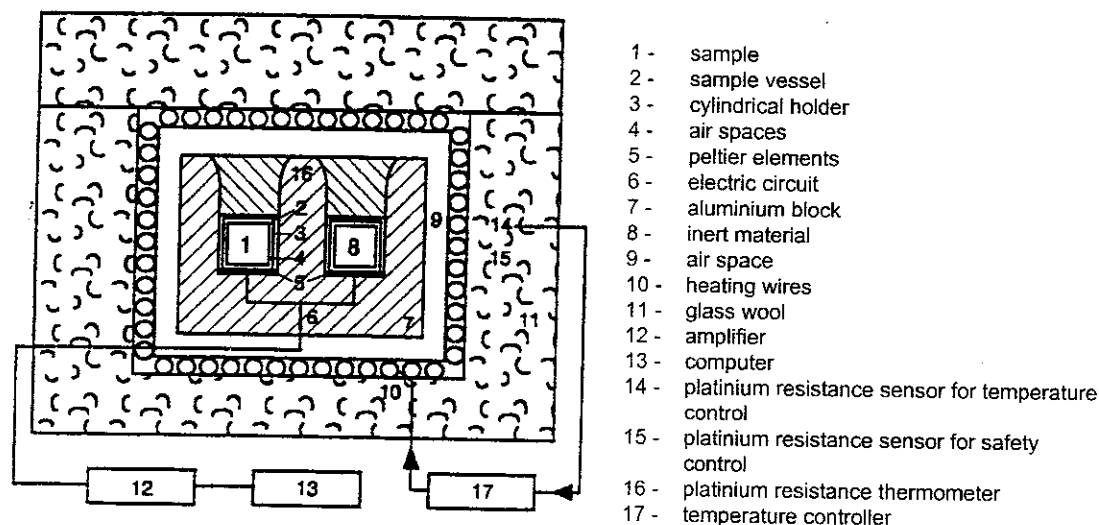


Fig.2-1. Schematic diagram of the Heat Flow Calorimeter.

4. PREPARATION OF EXPLOSIVES

With necessary precautions, grind, rasp or subdivide the explosive to pass through a sieve with a 2 mm opening. Discard material which passes a 0.2 mm sieve. When the explosive contains a major ingredient (such as TNT) which melts during the course of the test, a larger grist size is permissible. Explosives which do not require grinding or sub-division to pass a 2 mm sieve shall not be sieved before use to remove fine particles. The specimen shall be dried or prepared in accordance with the requirements for explosive under test.

5. PREPARATION OF TEST MATERIALS

The material used for the test shall be, as far as is practical, in its proposed condition for contact with the explosive. Where the intended use requires the test material to be applied in an intermediate (uncured or partly cured) condition to the explosive, the heat generation to bring the test material to its final (cured) condition.

Most solid materials require to be subdivided for testing to obtain effective mixing and a high degree of contact with the explosive during the test.

Subdivision of solid materials in block form must reduce them to particles with a maximum size of 2 mm and the preparation of materials as shavings or by cutting up of thin sheets or other means must allow a degree of mixing and contact with the explosive which is at least as effective. Discard material which has been produced by the process of subdivision and which passes a 0.2 mm sieve.

7. PRELIMINARY TEST

When testing materials of unknown chemical reactivity it is desirable to confirm by means of simple tests, in which small quantities are mixed with explosives and warmed, that reactions between the substances being tested for compatibility are not liable to cause explosion or fire during the test.

8. COMPATIBILITY TEST

- 8.1 Mix 2.5 g. of the test explosive with 2.5 g. of the test material and transfer the mixture to the heating tube of the apparatus, taking care to avoid contamination of the neck of the tube (the introduction of the explosive can be facilitated by the use of a wide-bore short-stemmed glass funnel). A minimum of two tests shall be conducted.
- 8.2 Twist the heating tube so that the holes in the joints correspond (for method 1 only). Verify that these are free of lubricant, slide the inner tube outwards (for method 2 only). Connect the assembly to the vacuum pump using a vacuum line and rubber adapter. Evacuate the assembly until the pressure is reduced to a maximum of 5 mm of mercury. Tap the heating tube lightly to facilitate release of any occluded air from the sample. Twist the heating tube carefully over 180° (for method 1 only), or slide the inner tube inwards (for method 2 only).
- 8.3 Record the voltmeter reading of the transducer at room temperature and check for any drift on the voltmeter (the transducer needs a 10 to 30 min. warming up period).
- 8.4 Record the voltmeter reading (R_1) and the room temperature at the beginning of the test (t_1 °C).
- 8.5 Ascertain that the bath temperature is constant at the specified temperature. Carefully place the heating tube of the prepared test assembly in the constant temperature bath.
- 8.6 Heat the tube for the specified heating period. Check the bath temperature every day.
- 8.7 Remove the tube from the constant temperature metal block. After allowing to cool at ambient temperature for not more than 2 hours 30 minutes (so as to limit the re-absorption of the gases evolved during the test) measure the amount of gas evolved.
- 8.8 Record the voltmeter reading (R_2) and the room temperature at the end of the test (t_2 °C).
- 8.9 Execute the same tests with:
 - An heating tube containing only 2.5 g of explosive.
 - An heating tube containing only 2.5 g of the test material.
 - Heat both samples using conditions identical to those used for heating the mixture.

9. CALCULATION

- 9.1 Using method 1 or 2 below, calculate the volume of gas V (at STP).
 - 9.1.1 Method N° 1 This method is applicable if the free volume has been calculated using method N° 1, paragraph 4.2.1.

Calculate the gas pressures P_1 and P_2 corresponding with the voltmeter readings R_1 and R_2 , using the calibration certificates mentioned in paragraph 4.1.2.

ANNEX D to
STANAG 4147
(Edition 2)

where:

- M = heat generation of the mixture of explosive and test material (J g^{-1})
E = heat generation of the explosive (J g^{-1})
S = heat generation of the test material (J g^{-1})

When $D > 3$: the test material must be considered as incompatible with the explosive.

When $2 < D < 3$: another method should be used to determine compatibility.

NOTE: The formula and criteria are only valid for an 1: 1 mixture of explosive and test material.

8.2 Other explosives:

Insufficient data are available to set criteria.

9. REFERENCE

- 9.1 An example of the set up of this test is described in "Recommendations on the Transport of Dangerous Goods, Tests and Criteria, ST/SG/AC.10/11/Rev.1" (the "Orange Book") of the United Nations.

- M = Volume of gas from 2.5 g of explosive mixed with 2.5 g of test material (cm^3 , at STP)
- E = Volume of gas from 2.5 g of explosive (cm^3 , at STP)
- S = Volume of gas from 2.5 g of test material (cm^3 , at STP)

- 9.4 Report the results of the test on the Data Sheet.
(Appendix 1 to Test 1B).

10. DISMANTLING AND CLEANING

Remove the apparatus from the heating metal block and release the vacuum. Dismantle the apparatus, empty the heating tube and remove residual traces of explosive from the apparatus with suitable solvents. Rinse the heating tube with acetone and then with water. Fill the tube with a suitable solution for glassware cleaning and allow to stand for 24 hours. Finally rinse with water followed by acetone and blow-dry with clean, dry air. Store the cleaned heating tubes in a desiccator until required for use.

Clean the pressure transducers according to the manufacturer's recommendations.

NATO STANAG 4147 DATA SHEET (Side B)	
Report Reference Number: (Unique Reference Number)	Page ___ of ___ Page(s)
DIVERGENCES FROM STANDARD PROCEDURE: (if applicable include details of drying, curing or solvent removal)	
TEST RESULTS: (Record the values of M, E, S and D. J g ⁻¹) Heat generation of the mixture of explosive and test material (M): Heat generation of explosive (E): Heat generation of test material (S): $\frac{2M}{(E + S)} = D$ Plot the heat generation rate curves, indicating the parameters used in the calculation of D. Attach the plot to the Data Sheet.	
COMPATIBLE: YES NO	
DATA SENT TO: (Name and address of person receiving this information)	
COMMENTS:	

TEST 1B

NATO STANAG 4147 DATA SHEET (Side B)	
Report Reference Number: (Unique Reference Number)	Page of Page(s)
DIVERGENCES FROM STANDARD PROCEDURE: (if applicable include details of drying, curing or solvent removal)	
TEST RESULTS: (Record the values of M, E, S and V_R cm ³ at STP)	
Volume of gas from 5 g of admixture (M):	
Volume of gas from 2.5 g of explosive (E):	
Volume of gas from 2.5 g of test material (S):	
$V_R = M - (E + S)$	
COMPATIBLE:	YES NO
DATA SENT TO: (Name and address of person receiving this information)	
COMMENTS:	

ANNEX D to
STANAG 4147
(Edition 2)

The difference in readings is the digital response for 2 cm³ of air at P mm and t °C. The response is determined from the two volumes of air to compensate for the "dead volume" between the syringe and adapter core. This calibrates the free space available in the apparatus after adding the test specimen to the heating tube. Repeat the calibration for every test.

Calculate the response factor for each heating tube and transducer, "f", for 1 cm³ air at STP:

$$f = \frac{(R_5 - R_3) - (R_3 - R_0)}{2} \times \frac{760}{P} \times \frac{(273 + t)}{273}$$

where:

- R₀ = empty tube voltmeter reading
 R₃ = voltmeter reading for 3.0 cm³ air
 R₅ = voltmeter reading for 5.0 cm³ air
 t = room temperature °C at the time of calibration of the apparatus
 P = atmospheric pressure (mm of mercury)

5. PREPARATION OF EXPLOSIVES

With necessary precautions, grind or subdivide the explosive to pass through a sieve with a 2 mm opening. Discard material which passes a 0.2 mm sieve. When the explosive contains a major ingredient (such as trinitrotoluene) which melts during the course of the test, a larger grist size is permissible. Explosives which do not require grinding or sub-division to pass a 2 mm sieve are not to be sieved before use to remove fine particles. The specimen shall be dried or prepared in accordance with the requirements for the explosive under test.

6. PREPARATION OF TEST MATERIALS

The material used for the test shall be, as far as is practical, in its proposed condition for contact with the explosive. When the intended use requires the test material to be applied in an intermediate (uncured or partly cured) condition to the explosive, the compatibility test shall be made on mixtures so prepared and then treated in the manner prescribed to bring the test material to its final (cured) condition.

Most solid materials require to be subdivided for testing to obtain effective mixing and a high degree of contact with the explosive during the test.

Subdivision of solid materials in block form must reduce them to particles with a maximum size of 2 mm and the preparation of materials as shavings or by cutting up of thin sheets or other means must allow a degree of mixing and contact with the explosive that is at least as effective. Discard material that has been produced by the process of subdivision and which passes a 0.2 mm sieve.

Test materials should not be artificially dried before testing but the presence of excessive quantities of volatile ingredients may make the test impossible to perform. To remove excessive quantities of water, preliminary drying over silica gel for 24 hours at ambient temperature is permitted. To remove volatile solvents other methods may be used. These treatments must be recorded in the test report.

TEST 1B

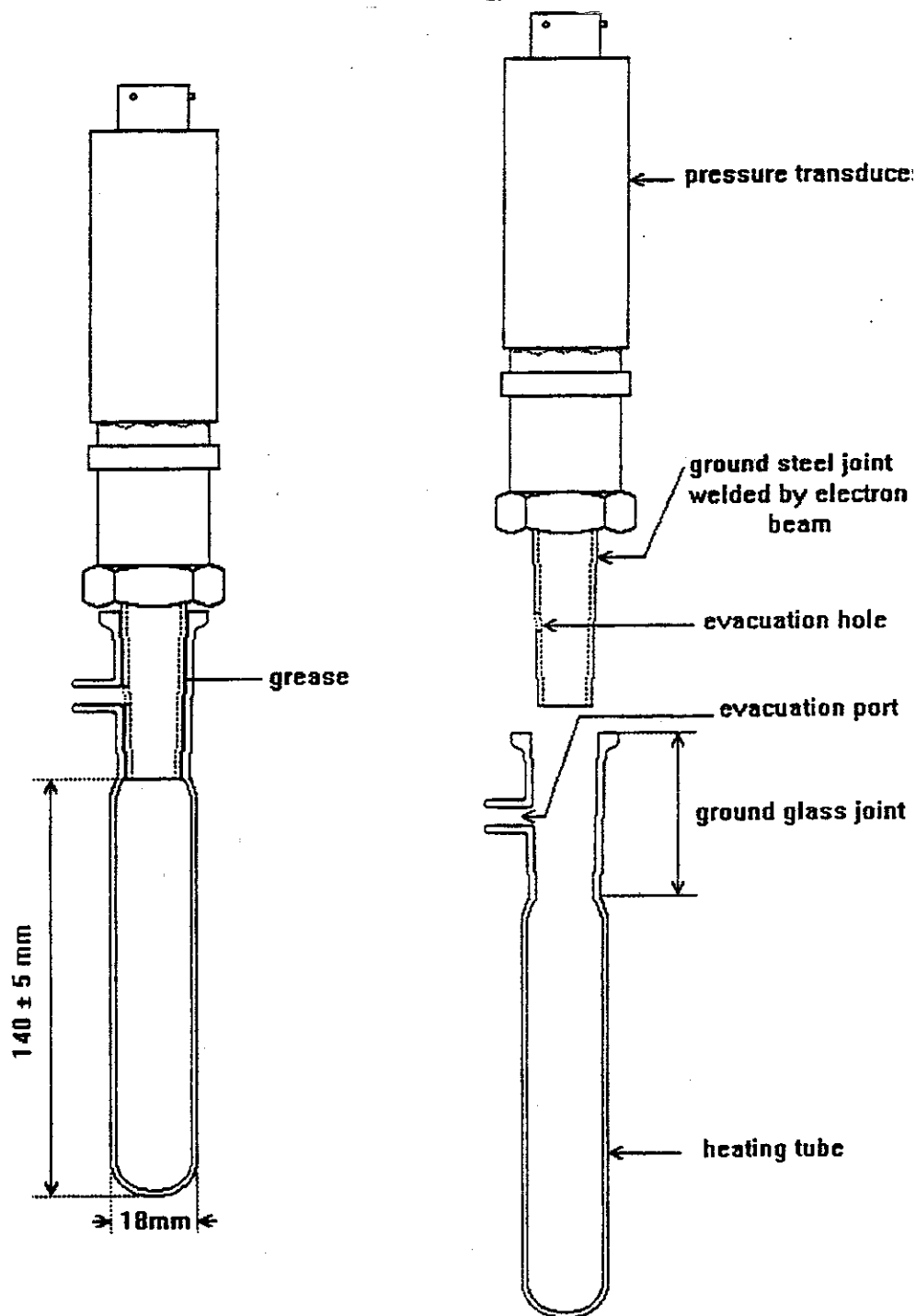


Figure 1B.1

ANNEX D to
STANAG 4147
(Edition 2)

Calculate the volume of gas V (at STP) liberated during the test, as follows:

$$V = \left(V_c + V_t - \sum \frac{m_i}{d_i} \right) \times \left[\frac{P_2 \times 273}{273 + t_2} - \frac{P_1 \times 273}{273 + t_1} \right] \times \frac{1}{1.013}$$

Where:

- V = volume of gas liberated from the sample (cm³, at STP)
- V_c = volume of the transducer and adapter (cm³)
- V_t = volume of the heating tube (cm³)
- P₁ = calculated pressure at the beginning of the test (bar).
- P₂ = calculated pressure at the end of the test (bar).
- t₁ = room temperature at the beginning of the test (°C)
- t₂ = room temperature at the end of the test (°C)
- m_i = mass of each substance tested (g)
- d_i = density of each substance tested (g cm⁻³)

- 9.1.2 Method N° 2 This method is applicable if the free volume has been calculated using method N° 2. Calculate the volume of gas, V, evolved from the specimen at STP:

$$V = \frac{R_2 \times (273 + t_1)}{f \times (273 + t_2)} - \frac{R_1}{f}$$

Where:

- R₁ = Voltmeter reading before heating
- R₂ = Voltmeter reading after the specified heating period
- t₁ = Room temperature °C at the time of calibration of the apparatus
- t₂ = Room temperature °C at the end of the test
- f = Response factor calculated in paragraph 4.2.2

- 9.2 Calculate, using the formula in method N° 1 or N° 2, the volumes of gas evolved by the mixture and the explosive and test material tested separately.

9.3 Calculate

$$V_R = M - (E + S)$$

Where:

- V_R = Volume of gas produced as a consequence of reaction between the components of the test mixture.

TEST 2THE HEAT FLOW CALORIMETRY TEST

The heat flow calorimetry test is a sensitive heat generation rate test at constant temperature. The amount of energetic material to be investigated is, typically, 1-5 gram and, therefore, the representativeness of the sample with respect to its chemical composition is better than, for instance, a screening test like the DSC.

For safety reasons the maximum amount of material to be used is 5 grams.

1. SUMMARY DESCRIPTION

The method determines the heat generation rate as a function of time produced by decomposing explosives at constant temperature. The heat release rate during isothermal storage simulates thermal aging for a certain period of time at a storage temperature. For propellants, storage for 168 hours at 85°C is a convenient test condition. Test time and test temperature shall be specified in the data sheet (Appendix 1).

The heat generation rate, as a function of time, of the mixture of explosive and test material, over a certain period of time is compared to a reference curve, which is the sum of the heat generation rates of the explosive and test material when heated separately. Acceptance criteria are given in Section 8.

2. APPLICABILITY

The heat flow calorimetry test is applicable to explosives, in particular nitrate ester based propellants. Due to insufficient data, criteria are not available for explosives other than ester based propellants.

The test can be performed under confined conditions using for example Helium or Nitrogen or under atmospheric conditions. As the results will depend on the test conditions, these conditions must resemble as closely as possible the practical situation and must be recorded in the data sheet (see Appendix 1).

3. APPARATUS

A brief description and diagram of a typical apparatus is given below:

The sample material is contained in a vessel (see Fig.2-1), made of material inert to the material to be tested, which is placed in close contact with a heat flow detector. A significant part of the heat generated by the sample passes through the heat flow detectors. Two identical measuring units (one for reference to increase sensitivity) of equal sensitivity are included symmetrically in the heat generation rate meter. The thermo-static measuring chamber is enclosed by a heat sink which itself is enclosed by a heating jacket.

The temperature of the heat sink is measured and then maintained to within $\pm 0.2^\circ\text{C}$ or better during the experiment.

The electric output of the heat flow detector can be amplified and recorded by a computer system.

NATO STANAG 4147 DATA SHEET (Side A)

Page ___ of ___ Page(s)

<div style="float: right; text-align: right;"> Page of Page(s) </div> TEST SITE INFORMATION		TEST CONDITIONS
Laboratory: (Name of Laboratory)	Test Temperature (°C):	
Date: (Date that form was completed)	Test Time (hours):	
Test Procedure: (Name of test procedure used)	Weight of samples:	
Date Tested: (Date of test period)	Test Material (g):	
POC: (Point of contact)	Test Explosive (g):	
	Test Admixture (1/1 g):	
SPECIMEN INFORMATION		SAMPLE PREPARATION
Identification of Test Material: (Trade name and/or Identity code)		Test Material, Preparation:
Manufacturer: (Name of Manufacturer)		Test Explosive, Preparation:
Test Material, Specification: (state if specification controls chemical composition of product)		
Lot, Batch or Consignment Number		
Date of Manufacture or Receipt:		
Special Storage Conditions: (if applicable)		
		TEST EXPLOSIVE
		Explosive Name:
		Date of Manufacture: (if known)
		Composition: (with percentages)

Test materials should not be artificially dried before testing but the presence of excessive quantities of volatile ingredients may make the test impossible to perform. To remove excessive quantities of water, preliminary drying over silica gel for 24 hours at ambient temperature is permitted. To remove volatile solvents other methods may be used. These treatments must be recorded in the data sheet.

6. PRELIMINARY TEST

When testing materials of unknown chemical reactivity it is desirable to confirm by means of simple tests in which small quantities of explosives and test material are mixed and heated, that reactions between the substances being tested for compatibility are not liable to cause explosion or fire during the test.

7. COMPATIBILITY TEST

The apparatus, including the reference sample vessel, shall be brought to the test temperature prior to inserting the sample vessel into the apparatus.

Before a measurement can be performed the blank signal and the sensitivity of the heat generation rate meter shall be determined.

NOTE: This can vary depending on the temperature used.

Determine the heat generation rate of a 1:1 mixture by weight. Mix 5 ± 0.01 grams of the test explosive with 5 ± 0.01 grams of the test material and transfer the mixture to the sample vessel. Close the sample vessel gastight (if required), then insert it into the heat generation rate meter.

After insertion of the sample vessel, temperature equilibration must be attained before the actual measurement can start.

NOTE: Less sample may be used, dependent on the apparatus used.

Measure the heat generation rate of the mixture for the period of 168 hours. Repeat the experiment under identical conditions, on the explosive and the test material separately. Discard the first five hours of the test period.

Determine the time integrated heat generation per unit of mass for the Mixture (M), the explosive (E) and the test material (S).

All tests shall be conducted in duplicate.

8. CRITERIA

8.1 Nitrate ester based propellants:

Calculate the relative increase in heat generation of the mixture of explosive and test material, compared to the heat generation of the components separately:

$$D = \frac{2M}{E + S}$$

Appendix 2 to
TEST 1B of
ANNEX D to
STANAG 4147
(Edition 2)

TEST 1B

ACCEPTANCE LIMIT FOR TEST 1B

1. For materials to which paragraph 2 of this Appendix does not apply, the volume of gas
 $V = M - (E + S)$ must not exceed 5 cm^3 at STP where:
 M = volume of gas evolved by 2.5 g of the test explosive when mixed with 2.5g of the test material and heated for specified heating period under the required test condition;
 E = volume of gas evolved by 2.5 g of the test explosive when heated for specified heating period under the required test condition;
 S = volume of gas evolved by 2.5 g of the test material when heated for specified heating period under the required test condition.
2. Materials supplied to specifications which include requirements for testing for compatibility with explosives or propellants by the Vacuum Stability Test, and for which the permitted volume of evolved gas V , as defined in paragraph 1 of this Appendix, is less than 5 cm^3 at STP must be tested and sentenced in accordance with the requirements of their specifications.
3. When V_R approaches 5 cm^3 , retest is recommended to confirm the result. Confirmation, using another test based on a different principle, should also be sought. If the second test is "failed", then the test material is not compatible with the explosive.

NATO STANAG 4147 DATA SHEET (Side A)

Page ___ of ___ Page(s)

NATO/PfP UNCLASSIFIED

Appendix 4 to
TEST 1B of
ANNEX D to
STANAG 4147
(Edition 2)

TEST 1B

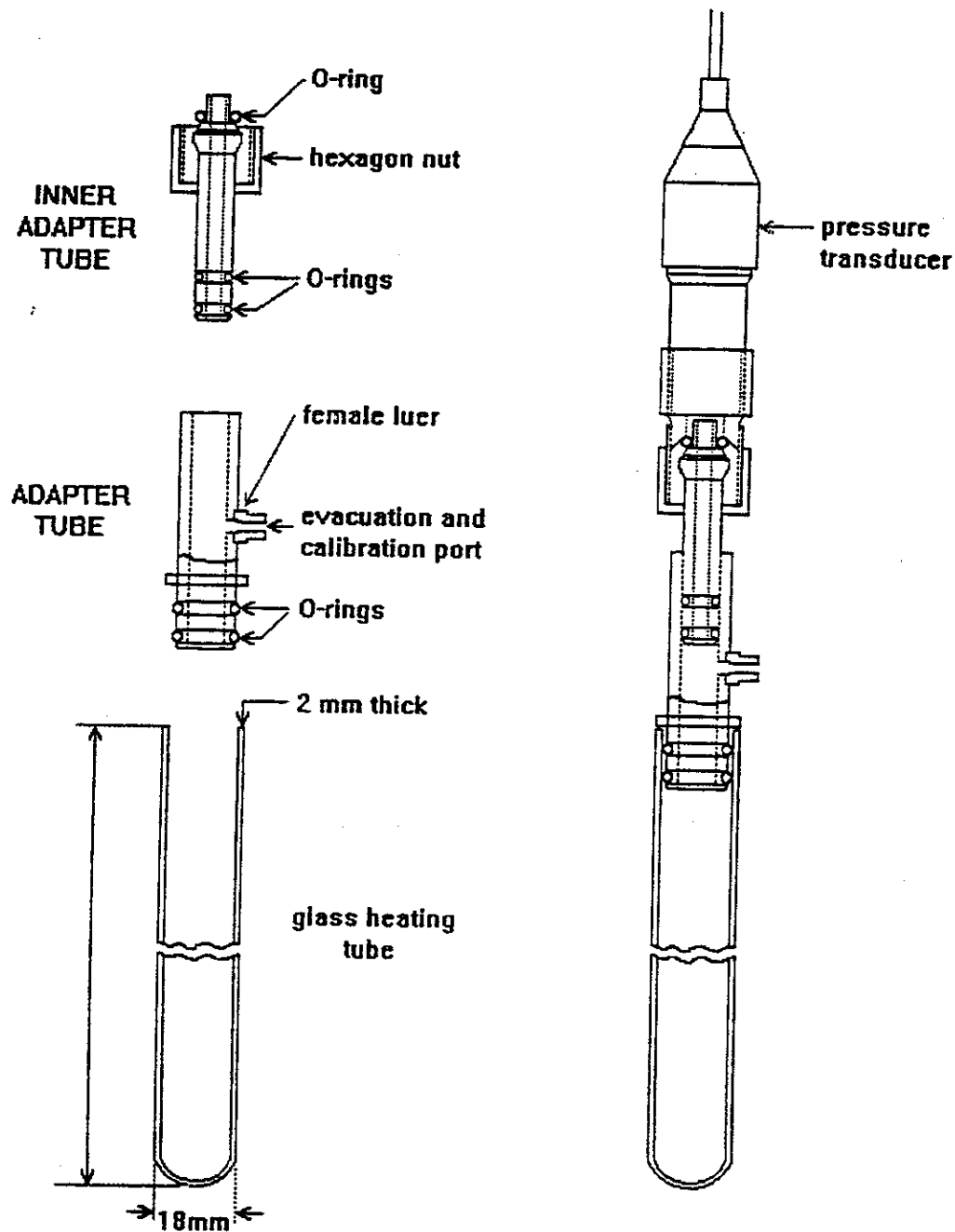


Figure 1B.2

TEST 3: PROCEDURE ADYNAMIC THERMOGRAVIMETRY (TGA)1. SUMMARY

Dynamic TGA is carried out at $2^{\circ}\text{C min}^{-1}$ in a nitrogen atmosphere on the individual explosives and test materials and on a mixture of the explosive and test materials in a 1:1 ratio by weight. The sum of the weight losses of the explosives and test materials is compared to that of a mixture of the explosives and test materials at a selected temperature. The temperature selected is the lowest exotherm reaction peak as determined by DSC, Test 4 or other suitable test methods. If the observed weight loss of the mixture is greater than the sum of the individual explosives and test materials then this is an indication of incompatibility. The greater the difference in percent weight loss the greater the degree of incompatibility at that temperature. The limits for the various degrees of incompatibility for TGA data in percent weight loss were chosen to correspond to the peak temperature shifts in DSC data.

2. APPLICABILITY

The test is applicable to explosives likely to come into contact with plasticizers, fuels, additives, and polymeric materials or other explosive materials.

3. SAMPLE PREPARATION

The material used for the test shall be, as far as is practical, in its proposed condition for contact with the explosive. When the intended use requires the test material to be applied in an intermediate (uncured or partially cured) condition to the explosive, the compatibility test shall be made on mixtures so prepared and then treated in the manner prescribed to bring the test material to its final (cured) condition.

Most solid materials require to be subdivided for testing to obtain effective mixing and a high degree of contact with the explosive during the test. Subdivision of solid materials in block form must reduce them to the smallest size particles as practical. The preparation of materials as shavings or by cutting up of thin sheets or other means must allow a degree of mixing and contact with the explosive that is effective.

Explosives can be subdivided for testing by grinding or rasping the explosive using the proper safety precautions. In some instances, applying pressure to the explosive once placed in the sample pan will cause it to break apart or spread so that sufficient surface area for contact with the test material has been achieved.

NOTE: Actual samples used in formulations are tested with the same particle size that would be used in the formulation. Reducing particle size may influence test results in this particular case. It is essential to ensure that the explosive is homogeneous so that the sample taken is representative of the composition.

Samples are prepared by placing a 1:1 (w/w) ratio of the test explosive and material in the sample pan. The sample weight of each component tested is about 1 mg. The explosive tested should be the same weight as used in the mixture or as close as possible. Samples are in physical contact with each other and not placed side-by-side. Specific placement of different matrices are as follows:

ANNEX D to
STANAG 4147
(Edition 2)

- 3.1 Non-volatile liquid and powdered sample: Solid is added first to the sample pan and the liquid placed on top.
- 3.2 Film/solid piece and powdered sample: Place the film in the pan first and add the powder to the top.
- 3.3 Two powdered/film samples: Place the sample which decomposes first on the bottom of the pan.

4. EXPERIMENTAL CONDITIONS

A heating rate of $2^{\circ}\text{C min}^{-1}$ and an anhydrous nitrogen atmosphere with a flow of $50 \text{ cm}^3 \text{ min}^{-1}$ is employed. The initial temperature selected is normally room temperature. However, if the thermal characteristics of the material are known, the test may be started at a higher temperature. TGA sample pans are inert, preferably of platinum construction. An aluminum liner made out of aluminum foil may be placed on the inside of the platinum sample pan to prolong the life of the platinum pan. The aluminum liner cannot be used above 600°C . Aluminum pans may also be used but, as with the liner, may cause problems with reactive materials. Any changes made to these conditions should be specified on the data sheet.

5. PROCEDURE

- 5.1. Each explosive and test material is run individually to determine the number of weight loss steps in the selected temperature range. The weight of the explosive and test material is plotted as a function of temperature. Plot the weight loss and the calculated derivative versus temperature. Select the derivative peak maximum.

NOTE: The term explosive refers to the finished explosive and may contain several different explosive ingredients. The finished explosive is treated as one test material unless there is a requirement to need to know which specific explosive ingredient is causing the incompatibility.

- 5.2 If the test material is another explosive, the two explosives are run individually and in contact with each other to determine the number of weight loss steps in the selected range. A derivative maximum peak temperature is selected for each explosive.
- 5.3 Admixtures are then prepared in a 1:1 ratio for explosive to non-explosive test materials. Weight losses are measured at the selected temperature corresponding to a weight loss step. The admixtures are run in duplicate.

When the admixture is a combination of two explosives, weight losses are measured at the two selected temperatures from step 5.2.

NOTE: If investigating multiple combinations, tests should be performed on every possible combination.

6. ANALYSIS

Record the data using the data sheet given in Appendix 2 page D 47.

- 6.1 For dynamic TGA, the difference between the observed weight loss and the total calculated weight loss of the explosive and test material in the admixture at a given temperature is noted. The temperature selected is either the derivative TGA peak or close to it. If the observed weight loss of the admixture is greater than that of the sum of the individual explosive/test material (total calculated), then this is an indication of incompatibility. The greater the difference between observed and calculated weight loss, the greater the degree of incompatibility. An example of this is as follows:

6.1.1 Observed weight loss at 200°C (derivative TGA peak)

Compound A (neat) weight loss = 10 %
Compound B (neat) weight loss = 5 %

The mixture consists of Compound A = 1.0 mg
Compound B = 0.8 mg

Therefore the % weight fraction of Compound A = 55.6 %
Compound B = 44.4 %

The observed weight loss of this admixture at 200°C = 18 %

6.1.2 Calculated weight loss

Compound A = (observed weight loss A) X (weight fraction A)
= (0.1) X (55.6) = 5.6 %

Compound B = (observed weight loss B) X (weight fraction B)
= (0.05) X (44.4) = 2.2 %

Total calculated weight loss equals 7.8%. (5.6% + 2.2%)

Since the observed weight loss of the admixture (18%) is greater than the calculated weight loss (7.8%), this sample shows a small degree of incompatibility.

If the observed weight loss of the admixture is less or equal than the sums of the individual explosive/test material (calculated weight loss), this indicates that the admixture is compatible. An example is as follows:

Observed weight loss at 200°C:

Compound A (neat) weight loss = 10 %
Compound B (neat) weight loss = 5 %

The mixture consists of Compound A = 1.0 mg
Compound B = 0.8 mg
Total weight = 1.8 mg

Therefore % weight fraction of Compound A = 55.6 %
Compound B = 44.4 %

The observed weight loss of this admixture at 200°C = 7.0 %

ANNEX D to
STANAG 4147
(Edition 2)

Calculated weight loss

$$\begin{aligned}\text{Compound A} &= (\text{observed weight loss A}) \times (\text{weight fraction A}) \\ &= (0.1) \times (55.6) = 5.6 \%\end{aligned}$$

$$\begin{aligned}\text{Compound B} &= (\text{observed weight loss B}) \times (\text{weight fraction B}) \\ &= (0.05) \times (44.4) = 2.2 \%\end{aligned}$$

Total calculated weight loss equals 7.8 %

Since the observed weight loss of the admixture (7.0 %) is less than the calculated weight loss (7.8 %), this admixture is compatible. No chemical reaction occurred between the explosive and test material, resulting in the formation of off gases and resultant increase in weight loss.

7. CRITERIA

- 7.1 A change of less than 4% by weight difference (% observed weight loss - % calculated weight loss) indicates the materials are compatible. A change between 4% and 20% indicates a degree of incompatibility may exist and further testing is required. A change greater than 20% by weight difference indicates incompatibility of the material and explosive under test.
- 7.2 A flowchart in Figure 3A.1 outlines the decision making process used to determine compatibility. When a material falls within the grey area of 4-20%, other suitable test methods should be conducted. Test 3 Procedure B outlines a more stringent application of TGA in the isothermal mode, and can be used as a confirmation test for the dynamic TGA procedure. Kinetics studies can also be used as a confirmation test method. The kinetic test procedure is Test 3 Procedure C. It should be noted that kinetic studies are extremely time consuming and should be only performed when other test methods are not suitable.

TEST 3 A

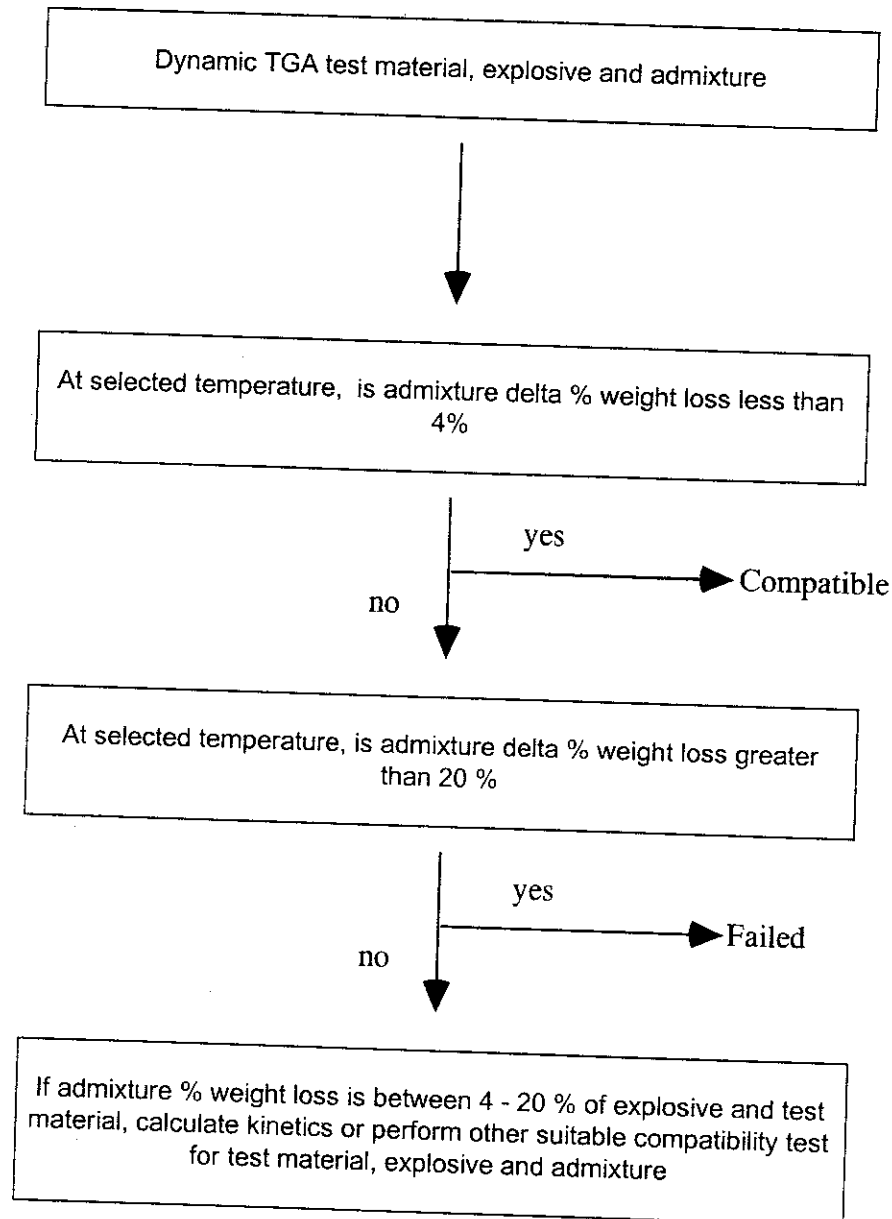


Fig. 3A.1: Thermogravimetry compatibility scheme using one test material and explosive

TEST 3: PROCEDURE B

ISOTHERMAL THERMOGRAVIMETRY (TGA)

1. **SUMMARY**

A second stringent TGA test for compatibility is heating the admixture, explosive and test material, at a specific temperature for 1000 minutes. The weight loss of the admixture, explosive and test material are compared after 1000 minutes at the temperature of interest, as specified by Test 3A. The same guidelines for degree of incompatibility as dynamic TGA are applied.

2. **APPLICABILITY**

This test is applicable to explosives likely to come in contact with plasticizers, fuels, additives, polymeric materials and other explosive materials.

3. **SAMPLE PREPARATION**

The material used for the test shall be, as far as is practical, in its proposed condition for contact with the explosive. When the intended use requires the test material to be applied in an intermediate (uncured or partially cured) condition to the explosive, the compatibility test shall be made on mixtures so prepared and then treated in the manner prescribed to bring the test material to its final (cured) condition.

Most solid materials require to be subdivided for testing to obtain effective mixing and a high degree of contact with the explosive during the test. Subdivision of solid materials in block form must reduce them to the smallest size particles as practical. The preparation of materials as shavings or by cutting up of thin sheets or other means must allow a degree of mixing and contact with the explosive that is effective.

Explosives can be subdivided for testing by grinding or rasping the explosive using the proper safety precautions. In some instances, applying pressure to the explosive once placed in the sample pan will cause it to break apart or spread so that sufficient surface area for contact with the test material has been achieved.

NOTE: Actual samples used in formulations are tested with the same particle size that would be used in the formulation. Reducing particle size may influence test results in this particular case. However, it is essential to ensure that the explosive is homogeneous so that the sample taken is representative of the composition.

Samples are prepared by placing a 1:1 (w/w) ratio of the test explosive and material in the sample pan. The sample weight of each component tested is about 1 mg. The explosive tested should be the same weight as used in the mixture or as close as possible. Samples are in physical contact with each other and not placed side-by-side. Specific placement of different matrices are as follows:

- 3.1 Non-volatile liquid and powdered sample: Solid material is added first to the sample pan and the liquid placed on top.
- 3.2 Film/solid piece and powdered sample: Place the film in the pan first and add the powder to the top.

- 3.3 Two powdered/film samples: Place the sample which decomposes first on the bottom of the pan.

4. EXPERIMENTAL CONDITIONS

Temperature is determined by Test 3A. Use the temperature of the first onset of exotherm corresponding to a weight loss or 20°C below the weight loss derivative peak. If test 3A indicates incompatibility between the explosive and test material, run the isothermal TGA at the temperature at which incompatibility was indicated by Test 3A. This temperature is held for 1000 minutes under an anhydrous nitrogen atmosphere with a flow rate of $50 \text{ cm}^3 \text{ min}^{-1}$.

5. PROCEDURE

- 5.1 Individual explosive and test materials are run isothermally at the set temperature to determine the number of weight loss steps for each explosive and test material. These weight loss steps are the derivative maximum TGA peaks for each material.

NOTE: The term explosive refers to the finished explosive and may contain several different explosive ingredients. This finished explosive is treated as one test material unless there is a requirement to know which specific explosive ingredient is causing the incompatibility.

- 5.2 Admixtures are then prepared at a 1:1 ratio and weight losses measured at the selected temperature.

6. ANALYSIS

The calculated and observed weight values are determined for each component and combination similar to the dynamic TGA procedure described in Test 3A.

7. CRITERIA

- 7.1 Any change under a 4 % by weight difference (% observed weight loss - % calculated weight loss) indicates the materials are compatible. Any change between 4% and 20% indicates a degree of incompatibility may exist and further testing is required. A greater than 20 % weight difference indicates incompatibility of the material and explosive under test.

- 7.2 A flow chart in Figure (3B.1) outlines the decision making process used to determine compatibility. When a material falls within the grey area of 4%-20%, other suitable test methods should be conducted. Kinetics studies can also be used as a confirmation test method. The kinetic test procedure is Test 3 Procedure C. It should be noted that kinetic studies are extremely time consuming and should only be performed when other test methods are not suitable.

TEST 3 B

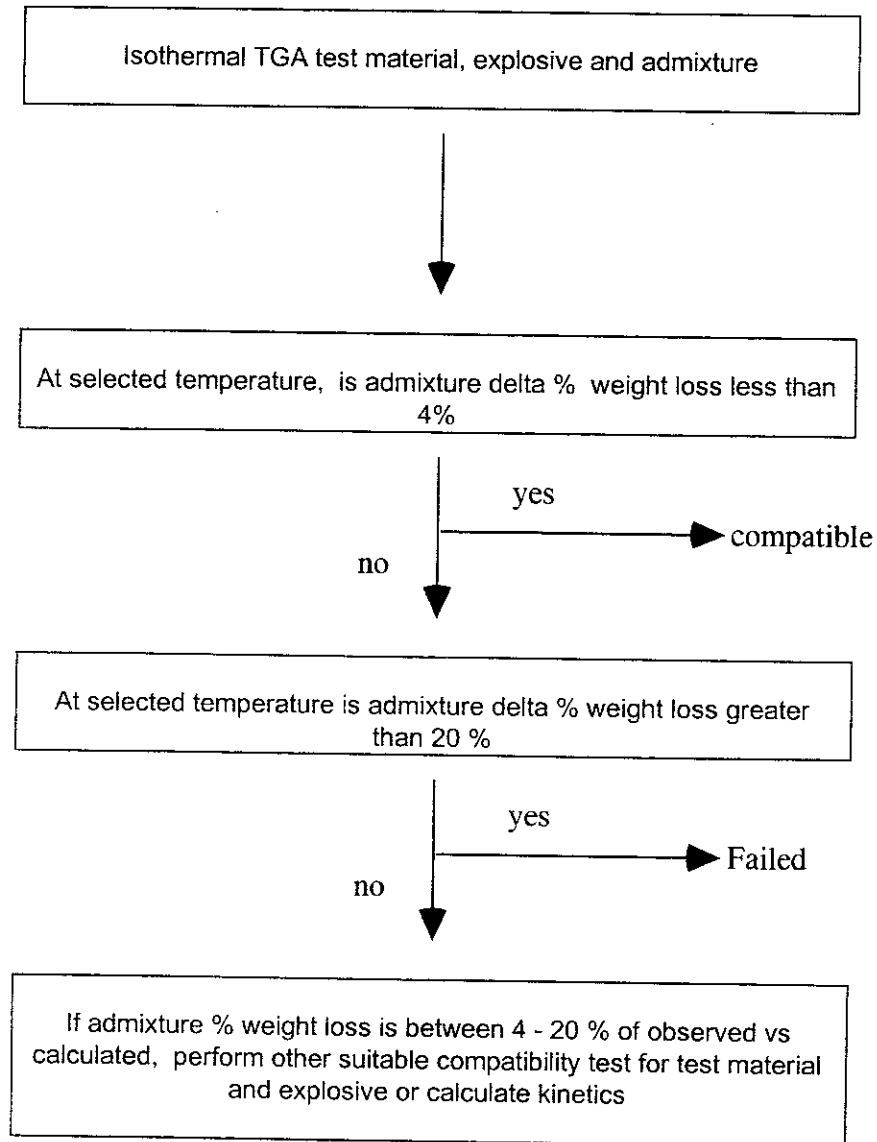


Fig. 3B.1: Isothermogravimetry compatibility scheme using one test material and explosive.

TESTS 3 A & B

NATO STANAG 4147 DATA SHEET (Side A)	
Report Reference Number: (Unique Reference Number):	
Page ____ of ____ Page(s)	
TEST SITE INFORMATION Laboratory: (Name of Laboratory) Date: (Date that form was completed) Test Procedure: (Name of test procedure used) Date Tested: (Date of test period) POC: (Point of contact)	TEST CONDITIONS Sample atmosphere: Heating Rate: Temperature Range: Type of TGA Pan: Divergence from Std Procedure:
SPECIMEN INFORMATION Identification of Test Material: (Trade name and/or Identity code) Manufacturer: (Name of Manufacturer) Test Material, Specification: (state if specification controls chemical composition of product) Lot, Batch or Consignment Number: Date of Manufacture or Receipt: Special Storage Conditions: (If applicable)	SAMPLE PREPARATIONS Method of Preparation of Test Material: Sample Preparation Weights: Sample A, Test Material (mg): Sample B, Explosive (mg): Sample M, Admixture 1/1 (mg):
	TEST EXPLOSIVE Explosive Name: Date of Manufacture: (if known) Composition: (with percentage)

Appendix 2 to
TEST 3A & 3B of
ANNEX D to
STANAG 4147
(Edition 2)

TEST 3 A & B

NATO STANAG 4147 DATA SHEET (Side B)	
Report Reference Number:	
(Unique Reference Number):	
Page ____ of ____ Page(s)	
TGA TEMPERATURE °C	CALCULATIONS
Derivative Peak Temperature, °C:	% Weight Fraction Sample A
Observed Weight loss, Wt%:	% Weight Fraction Sample B
Sample A, neat:	Calculated Weight Loss, A
Sample B, neat:	Calculated Weight Loss, B
Admixture, M:	Calculated Total Weight Loss, D
	M - D =
OBSERVATIONS/COMMENTS	RESULTS
	Material Compatible, Y/N:
	Confirmation Test Required, Y/N:
	RESULTS SENT TO:

TEST 3: PROCEDURE C**DETERMINATION OF THE KINETICS OF DECOMPOSITION****1. SUMMARY**

Thermal compatibility determinations are not ideal because they take place at elevated temperatures i.e., > 100 °C, in order to detect changes in physical properties that can be measured by DSC and TGA. Incompatibility should be determined at room temperature. Kinetic analysis requires determining the activation energy (E), Arrhenius frequency factor (A), and calculating the decomposition rate constant (k) for the admixture, explosive and test material. This can be conducted using a variable heating rate method.

DSC and derivative TGA (dTGA) curves are determined for samples using three linear heating rates and recording any exothermic reaction peaks. The kinetic values of E, A and k are calculated from the peak temperature and heating rates following the Kissinger method (Ref. 8-1 and 8-2)

If the test material lowers the activation energy, increases the frequency factor or rate of reaction for decomposition of the admixture, then the test material is incompatible. The reaction rate constant at room temperature may be calculated from the equation: $\ln k = \ln A - (E/RT)$. However, it must be assumed that the mechanism of reaction does not change with change in temperature i.e., that E and A are invariant. If the rate constant for the admixture is significantly higher (a thousand fold) then the test material is likely to be incompatible at room temperature.

The kinetic constants obtained by the variable heating rate method must be verified by performing a 60 minutes half-life determination (see Section 6.8 for details). The enthalpy is determined and must agree to within +/- 5 % of the calculated value.

The determination of the kinetics of decomposition is very labour intensive. It is suggested that whenever possible other compatibility tests, as described in this STANAG, are performed to confirm the compatibility results of Tests 3A and 3B. Test 3C should only be undertaken when either kinetic data is required or compatibility of a material cannot be confirmed by any other means.

2. APPLICABILITY

This test is applicable to explosives likely to come in contact with plasticizers, fuels, additives, polymeric materials and other explosive materials.

3. SAMPLE PREPARATION

The material used for the test shall be, as far as is practical, in its proposed condition for contact with the explosive. When the intended use requires the test material to be applied in an intermediate (uncured or partially cured) condition to the explosive, the compatibility test shall be made on mixtures so prepared and then treated in the manner prescribed to bring the test material to its final (cured) condition.

ANNEX D to
STANAG 4147
(Edition 2)

Most solid materials require to be subdivided for testing to obtain effective mixing and a high degree of contact with the explosive during the test. Subdivision of solid materials in block form must reduce them to the smallest size particles as practical. The preparation of materials as shavings or by cutting up of thin sheets or other means must allow a degree of mixing and contact with the explosive that is effective.

Explosives can be subdivided for testing by grinding or rasping the explosive using the proper safety precautions. In some instances, applying pressure to the explosive once placed in the sample pan will cause it to break apart or spread so that sufficient surface area for contact with the test material has been achieved.

Note: Actual samples used in formulations are tested with the same particle size that would be used in the formulation. Reducing particle size may influence test results in this particular case. However, it is essential to ensure that the explosive is homogeneous so that the sample taken is representative of the composition.

Samples are prepared by placing a 1:1 (w/w) ratio of the test explosive and material in the sample pan. The sample mass of each component tested is about 1 mg. The explosive tested should be the same weight as used in the mixture or as close as possible. Samples are in physical contact with each other and not placed side-by-side. Specific placement of different matrices are as follows:

- 3.1 Non-volatile liquid and powdered sample: Solid is added first to the sample pan and the liquid placed on top.
- 3.2 Film/solid piece and powdered sample: Place the film in the pan first and add the powder to the top.
- 3.3 Two powdered/film samples: Place the sample which decomposes first on the bottom of the pan.

4. EXPERIMENTAL CONDITIONS

- 4.1 The sample container used must be non-reactive with the sample and/or reaction products. Coated aluminum pans or inert platinum pans can be used. When performing DSC, if the sample has any volatility over the temperature range of interest, the pan should be hermetically sealed to prevent vaporization. If the pan balloons then the amount of sample should be reduced or a perforated sample pan cover can be used in an inert atmosphere.
- 4.2 The sample atmosphere should closely resemble the conditions of usage. When performing TGA, an anhydrous nitrogen atmosphere is used with a flow rate of $50 \text{ cm}^3 \text{ min}^{-1}$.

5. PROCEDURE

- 5.1 Run each individual explosive, test material and admixture at a heating rate of $0.2^{\circ}\text{C min}^{-1}$. The starting temperature should be ambient or at least 50°C below the first observed exothermic peak. Continue heating through the decomposition peak until the curve returns to or near the baseline that was observed before the decomposition peak. Generally the first decomposition peak or weight loss is the peak of interest. This may or may not be due to the energetic ingredient.
- 5.2 Repeat step 5.1 using at least three other heating rates between 0.2 and $2^{\circ}\text{C min}^{-1}$. A minimum of three heating rates is required. Suggested heating rates are 0.5 , 1 , and $2^{\circ}\text{C min}^{-1}$. In the case of explosives with high activation energies, it may be necessary to expand the heating rate range.

6. ANALYSIS

- 6.1 Temperatures of reaction peak maxima are recorded for each explosive, test material and admixture. Temperatures should be corrected for temperature scale nonlinearity, heating rate changes, and thermal lag according to instrument manuals.
- 6.2 Plot $\ln(B/T^2)$ versus $1/T$. B is the heating rate in Kmin^{-1} and T is the corrected peak maximum in K .
- 6.3 The activation energy can be determined from the slope, S , of the line according to the following equation:

$$E = -SR$$

where:

$E =$ Activation energy (J mole^{-1})

$R =$ Gas constant ($8.3143 \text{ J K}^{-1} \text{ mole}^{-1}$)

$S =$ Slope

- 6.4 The Arrhenius frequency factor, A (min^{-1}), can be calculated from the following equation. B used in this equation is the heating rate from one of the middle heating rates.

$$A = \frac{BEe^{(E/RT)}}{RT^2}$$

- 6.5 Other methods of calculation may be used (Ref 8-2).
- 6.6 The specific reaction rate constant, k , can then be determined using the Arrhenius equation,

$$k = Ae^{-E/RT}$$

The value of k should be calculated for all three temperatures for each explosive, test material and admixture.

- 6.7 Calculate the half-life times of reaction for k values using the following equation which assumes a first order reaction:

$$t_{1/2} = 0.693/k$$

An example is as follow:

Set 60 minutes $= t_{1/2}$

ANNEX D to
STANAG 4147
(Edition 2)

Then $k = 0.693/60 = 0.1155 \text{ min}^{-1}$

The rate for 60 minutes is then 0.11555 min^{-1}

Substitute into the equation $\log k = \log A - E/2.3RT$ and solve for T.

- 6.8 Age a sample isothermally at the calculated temperature for 60 minutes. Remove the sample and cool to ambient temperature. Allow the DSC or the TGA to cool to room temperature. Repeat this test a minimum of 5 times because the integration is performed on a peak that has a mass which is decreasing due to decomposition.

- 6.9 Run each of the five aged samples and an unaged sample under the same temperature and heating rate conditions.

NOTE: Five integrations are not necessary when using TGA and because of the higher precision of TGA, only two aged samples need to be examined.

- 6.10 The peak area or displacement from the baseline of the aged sample should be 1/2 that of the unaged sample. Integration of the peak areas must be performed at exactly the same temperature range used to determine the original k value. The five integrations are averaged if DSC is used. Reaction kinetics are confirmed if observed results are within +/- 5 % of the calculated values.

7. CRITERIA

- 7.1 The rate constant, frequency factor and activation energy of the admixture and test material are compared. The following criteria applies when interpreting the data.
- 7.2 If the activation energy is lower and the frequency factor is larger for the admixture than the explosive, then the test material is considered incompatible.
- 7.3 If only small differences in activation energy and frequency factors are observed but the rate constant of the admixture is greater than the explosive at room temperature, then the test material is considered incompatible.
- 7.4 However small the difference in rate constants, if the value for the admixture at the temperature of interest is equal to or less than 10, then there is a strong indication that the test material is compatible.
- 7.5 If the temperature of interest is elevated (i.e. > 200°C) then the rate constant would be much lower at room temperature and would be compatible.

8. REFERENCES

- 8.1 Two references for this method are 1) H.E. Kissinger, Analytical Chem. 29, 1702 (1957) and 2) H.E. Kissinger, J. Res. NBS 57, 217 (1956).
- 8.2 Other methods of calculations can be found in 3) ASTM E698-73.

TEST 3C DATA SHEET
WORKSHEET A

TEST 3C DATA SHEET WORKSHEET A																															
Report Reference Number: (Unique Reference Number):																															
Page ____ of ____ Page(s)																															
TEST SITE INFORMATION Laboratory: (Name of Laboratory) Date: (Date that form was completed) Test Procedure: (Name of test procedure used) Date Tested: (Date of test period) POC: (Point of contact)	TEST CONDITIONS Sample Weights: Sample A, test material (mg): Sample B, explosive (mg): Mixture M 1/1 (mg): Type Sample Pan: Atmosphere (Static flow): Heating Rate: Peak Temperature Range: Divergence from Std Test Conditions:																														
SPECIMEN INFORMATION Identification of Test Material: (Trade name and/or identity code) Manufacturer: (Name of Manufacturer) Test Material, Specification: (state if specification controls chemical composition of product) Lot, Batch or Consignment Number: Date of Manufacture or Receipt: Special Storage Conditions: (If applicable)	TEST DATA Reactive Peak Maximum: <table border="1"> <thead> <tr> <th></th> <th>Heating Rate (K.min⁻¹)</th> <th>Peak Maximum Temp. (K)</th> </tr> </thead> <tbody> <tr> <td>Sample A</td> <td>_____</td> <td>_____</td> </tr> <tr> <td></td> <td>_____</td> <td>_____</td> </tr> <tr> <td></td> <td>_____</td> <td>_____</td> </tr> <tr> <td>Sample B</td> <td>_____</td> <td>_____</td> </tr> <tr> <td></td> <td>_____</td> <td>_____</td> </tr> <tr> <td></td> <td>_____</td> <td>_____</td> </tr> <tr> <td>Mixture M</td> <td>_____</td> <td>_____</td> </tr> <tr> <td></td> <td>_____</td> <td>_____</td> </tr> <tr> <td></td> <td>_____</td> <td>_____</td> </tr> </tbody> </table>		Heating Rate (K.min ⁻¹)	Peak Maximum Temp. (K)	Sample A	_____	_____		_____	_____		_____	_____	Sample B	_____	_____		_____	_____		_____	_____	Mixture M	_____	_____		_____	_____		_____	_____
	Heating Rate (K.min ⁻¹)	Peak Maximum Temp. (K)																													
Sample A	_____	_____																													
	_____	_____																													
	_____	_____																													
Sample B	_____	_____																													
	_____	_____																													
	_____	_____																													
Mixture M	_____	_____																													
	_____	_____																													
	_____	_____																													
TEST EXPLOSIVE Explosive Name: Date of Manufacture or Receipt: (if known) Composition: (with percentage)																															
CALCULATIONS: Slope, S: Activation Energy, E: Arrhenius Freq. Factor, A: Reaction Rate Constant, k for: Test Material _____ Explosive _____ Admixture _____	TEST RESULTS Compatibility, Y/N: Half-Life Test Performed, Y/N: Confirmation, Y/N:																														
DATA SENT TO:																															

Appendix 1 to
TEST 3C of
ANNEX D to
STANAG 4147
(Edition 2)

TEST 3C DATA SHEET**WORKSHEET B**

TEST 3C DATA SHEET																		
WORKSHEET B																		
CALCULATIONS FOR HALFLIFE TEST																		
Report Reference Number: (Unique Reference Number):		Page ___ of ___ Page(s)																
TEST CONDITIONS Sample Weights: Sample A, test material (mg): Sample B, explosive (mg): Mixture M 1/1 (mg): Type Sample Pan: Atmosphere (Static Flow): Heating Rate: Peak Temperature Range: Divergence from Std Test Conditions:	TEST DATA Peak Integration: <table border="1"> <thead> <tr> <th>Aged</th> <th>Unaged</th> </tr> </thead> <tbody> <tr><td>_____</td><td>_____</td></tr> <tr><td>_____</td><td>_____</td></tr> <tr><td>_____</td><td>_____</td></tr> <tr><td>_____</td><td>_____</td></tr> <tr><td>_____</td><td>_____</td></tr> <tr><td>_____</td><td>_____</td></tr> <tr><td>_____</td><td>_____</td></tr> </tbody> </table> Average		Aged	Unaged	_____	_____	_____	_____	_____	_____	_____	_____	_____	_____	_____	_____	_____	_____
Aged	Unaged																	
_____	_____																	
_____	_____																	
_____	_____																	
_____	_____																	
_____	_____																	
_____	_____																	
_____	_____																	
CALCULATIONS: $t_{1/2}$ calc: Av peak integration, aged: (1) Av peak integration, unaged: (2) (2) - (1) =	TEST RESULTS Observed within +/- 5 % of calculated, Y/N:																	

TEST 4DIFFERENTIAL SCANNING CALORIMETRY1. SUMMARY DESCRIPTION

The DSC sample is contained either in a crimped, sealed pan with a pin hole in the lid or in a coated hermetically sealed pan in order to determine if the evolved gases affect the decomposition reaction. The peak temperatures of decomposition of the admixture, explosive and test materials are noted. If the peak due to the admixture is shifted to a lower temperature than the peaks due to the explosive and test materials, then this is an indication of a degree of incompatibility. As the temperature difference between the admixture, explosive and test material peaks increases so does the degree of incompatibility.

2. APPLICABILITY

This test is applicable to explosives likely to come into contact with plasticizers, fuels, additives, polymeric materials and other explosives.

3. SAMPLE PREPARATION

The material used for the test shall be, as far as is practical, in its proposed condition for contact with the explosive. When the intended use requires the test material to be applied in an intermediate (uncured or partially cured) condition to the explosive, the compatibility test shall be made on mixtures so prepared and then treated in the manner prescribed to bring the test material to its final (cured) condition.

Most solid materials require to be subdivided for testing to obtain effective mixing and a high degree of contact with the explosive during the test. Subdivision of solid materials in block form must reduce them to the smallest size particles as practical. The preparation of materials as shavings or by cutting up of thin sheets or other means must allow a degree of mixing and contact with the explosive that is effective.

Explosives can be subdivided for testing by grinding or rasping the explosive using the proper safety precautions. In some instances, applying pressure to the explosive once placed in the sample pan will cause it to break apart or spread so that sufficient surface area for contact with the test material has been achieved.

NOTE: Actual samples used in formulations are tested with the same particle size that would be used in the formulation. Reducing particle size may influence test results in this particular case. It is essential to ensure that the explosive is homogeneous so that the sample taken is representative of the composition.

Samples are prepared by placing a 1:1 (w/w) ratio of the test explosive and material in the sample pan. The sample weight of each component tested is about 1 mg. The explosive tested should be the same weight as used in the mixture or as close as possible. Samples are in physical contact with each other and not placed side-by-side. Specific placement of different matrices are as follows:

ANNEX D to
STANAG 4147
(Edition 2)

- 3.1 Non-volatile liquid and powdered sample: solid is added first to the sample pan and the liquid placed on top.
- 3.2 Film/solid piece and powdered sample: place the film in the pan first and add the powder to the top.
- 3.3 Two powdered/film samples. Place the sample which decomposes first on the bottom of the pan.

4. EXPERIMENTAL CONDITIONS

A heating rate of $2^{\circ}\text{C min}^{-1}$ is used. The initial temperature shall range from room temperature to 300°C or more for each sample. Aluminum sample pans are coated and hermetically sealed. Preforated pans can also be used in an inert atmosphere. The pans are purchased pre-coated with a fluoride coating or other suitable material. The coating is designed to keep moisture from reacting with the aluminum pan. Other pans may also be used in place of coated pans. Any changes made to these conditions should be specified on the data sheet.

5. PROCEDURE

- 5.1 Decomposition profiles are run on each individual material following the experimental conditions.
- 5.2 Maximum peak temperatures are determined for the primary reaction peak.
- 5.3 Explosive and test materials are combined in a 1:1 ratio and decomposition profiles determined. A matrix can be used if an investigation requires more than two materials to be tested.
- 5.4 Maximum peak temperatures of the resulting exotherms of the combinations are determined.

NOTE: A number of exotherms may be obtained.

- 5.5 A DSC for each individual explosive, test material and admixture shall be run in duplicate as a minimum. The average of duplicate runs shall be used in the calculations.

6. ANALYSIS

- 6.1 Shifts in the peak temperature of a single exotherm, (T_d), corresponding to the decomposition of the explosive are examined. A shift in this peak temperature indicates an interaction between the explosive and test material. If the shift of the peak is towards a lower temperature, this indicates that the presence of the test material has accelerated the decomposition of the explosive. Any combination that lowers the peak temperature of the explosive peak has a degree of incompatibility. The greater the shift towards lower temperatures, the greater the degree of incompatibility of the combination. If two explosive materials are tested together, the exotherm peaks for both explosives are examined for any occurring shifts.
- 6.2 Calculation of the shift in peak temperatures can be determined by subtracting the peak temperature of the explosive and test material from the mixture peak temperature and is referred to as the delta.

NOTE: For those materials that undertake a phase change and also demonstrate a marked shift in the reaction peak of interest, further investigation is necessary.

- 6.3 Peak shapes must also be examined qualitatively for any changes that may occur due to an interaction between explosive and test materials. Specific changes to look for include:
 - 6.3.1 Evidence of a peak becoming narrower (peak height to half-width)
 - 6.3.2 Peak forms a loop or leans towards a higher temperature
 - 6.3.3 Appearance or disappearance of new or old peaks.

7. CRITERIA

- 7.1 Any delta of four or less indicates the test material is compatible. If the shift in peak temperature is greater than 4°C then the test material is considered incompatible.
- 7.2 The appearance of additional peaks or disappearance of old peaks may indicate incompatibility and further investigations must be undertaken.
- 7.3 Changes in peak shape may indicate incompatibility and further investigations must be undertaken.
- 7.4 A flow chart has been developed to follow the process of decisions required when determining whether a sample is compatible or not. Figure 4.1 lists a flow diagram which describes the decision process when one test material and one explosive are tested for compatibility using DSC.

NOTE: A shift in peak temperature of the admixture of between 4°C and 20°C below the original peak temperatures of the explosive and test materials indicates a degree of incompatibility. If test data falls into this "grey area" it may be necessary to obtain supporting data from alternative compatibility tests. Kinetic information can be used to provide specific information and procedures are outlined in Test 3C. However, it should be noted that reaction kinetic tests are extremely time consuming and other tests described in this STANAG may be more suitable for confirming compatibility.

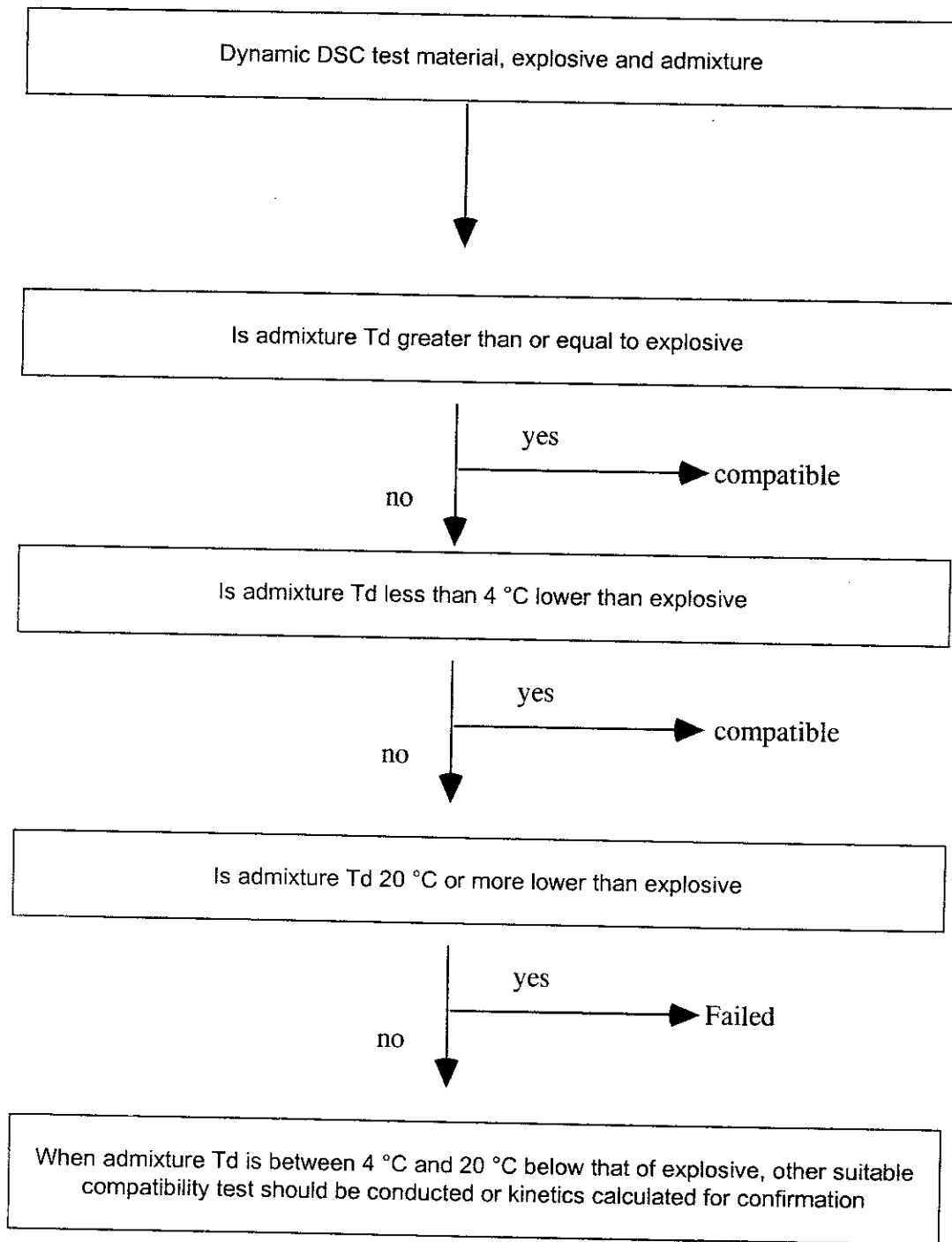
TEST 4

Fig. 4.1: DSC compatibility scheme using one ingredient and explosive

TEST 4

TEST 4A DATA SHEET FOR DSC METHOD (Side A)	
Report Reference Number: (Unique Reference Number):	
Page ____ of ____ Page(s)	
TEST SITE INFORMATION Laboratory: (Name of Laboratory) Date: (Date that form was completed) Test Procedure: (Name of test procedure used) Date Tested: (Date of test period) POC: (Point of contact)	TEST CONDITIONS Sample atmosphere: Heating Rate: Temperature Range: Type of TGA Pan: Divergence from Std Procedure:
SPECIMEN INFORMATION Identification of Test Material: (Trade name and/or identity code) Manufacturer: (Name of Manufacturer) Test Material, Specification: (state if specification controls chemical composition of product) Lot, Batch or Consignment Number: Date of Manufacture or Receipt: Special Storage Conditions: (if applicable)	SAMPLE PREPARATION Method of Preparation of Test Material: Sample Preparation Weights: Sample A, test material (mg): Sample B, explosive (mg): Sample Admixture M 1/1 (mg): TEST EXPLOSIVE Explosive Name: Date of Manufacture: (if known) Composition: (with percentage)

Appendix 2 to
TEST 4 of
ANNEX D to
STANAG 4147
(Edition 2)

TEST 4

TEST 4A DATA SHEET FOR DSC METHOD (Side B)			
Report Reference Number: (Unique Reference Number):		Page ____ of ____ Page(s)	
TEST DATA		OBSERVATIONS	
Sample	Exotherm Peak, °C	Peak Shape/Changes Observed	
A	_____		
B	_____		
M	_____		
DSC Peak Delta, °C: (mixture, M - explosive, A)			
RESULTS		DATA SENT TO:	
Compatibility, Y/N:			
Confirmation Test Required, Y/N:			

TEST 5

CHEMICAL ANALYSIS

INTRODUCTION

This procedure gives details of methods for artificially aging explosives in contact or close proximity to materials proposed for use as ammunition components. It also sets out guidelines on the type of chemical analysis required to determine the extent of degradation of the explosives after aging.

Artificial aging and chemical analysis is normally used for the assessment of compatibility with primary explosives and nitrate ester based propellants. Procedures A and B describe the methods for assessing nitrate ester based propellants and lead and silver azide respectively.

ANNEX D to
STANAG 4147
(Edition 2)

TEST 5 - CHEMICAL ANALYSIS PROCEDURE A

ASSESSMENT OF THE COMPATIBILITY OF AMMUNITION COMPONENT MATERIALS WITH NITRATE ESTER BASED PROPELLANTS

1. SUMMARY DESCRIPTION

The percentage stabilizer remaining, when a propellant sample in contact with a 10 % addition of the material under test is heated at a constant elevated temperature for 336 hours (14 days), is compared with the percentage stabilizer in an unheated sample and a sample heated under the same test conditions but in the absence of test material. Compatibility is judged by means of the additional loss of stabilizer because of contact with the material under test.

2. APPLICABILITY

The compatibility test is used for determining the reactivity of materials of ammunition components with nitrate ester based propellants.

3. INTERPRETATION OF RESULTS

Materials which fail to satisfy the requirements of this test will be judged as unsuitable for use in applications where permanent direct contact or close proximity with the test propellant is required or where adequate precautions to guard against such contact are not taken.

4. APPARATUS

4.1 One of the following, or a suitable equivalent, may be used to heat the propellant samples.

4.1.1 A constant temperature oven capable of maintaining a specified temperature to $\pm 0.2^{\circ}\text{C}$ over a period of 336 hours (14 days).

4.1.2 An electrically heated metal block bath capable of maintaining a specified temperature to $\pm 0.2^{\circ}\text{C}$ over a period of 336 hours (14 days). The block shall contain drilled holes able to accommodate the total length of the sample heating tubes. The diameter of the holes shall not be more than 2 mm greater than the diameter of the heating tube. An electrical cut-out shall be fitted that operates at 5°C above the nominal working temperature.

4.2 Glass heating tube of uniform bore, 25 mm x 152 mm with a ground glass joint capable of accepting a ground glass stopper, as illustrated in Appendix 3.

5. PREPARATION OF PROPELLANT TEST SAMPLE

Where appropriate and with necessary precautions, grind, rasp or subdivide the propellant to pass through a sieve with a 2.0 mm opening. Discard material which passes a 0.2 mm sieve. Propellants which do not require grinding or sub-division to pass a 2 mm sieve are not to be sieved before use to remove fine particles.

Propellant shall not be artificially dried before testing.

6. PREPARATION OF MATERIAL TEST SAMPLE

The material used for the test shall be, as far as is practicable, in the condition proposed for contact with the propellant. Where the intended use requires the test material to be applied in an intermediate (uncured or partly cured) condition to the propellant, the compatibility test shall be made on mixtures so prepared.

Most solid materials require to be subdivided for testing to obtain effective mixing and a high degree of contact with the propellant during the test.

Wherever possible, grind, rasp or cut solid material to particles with a maximum size of 2 mm. Discard material which has been produced by the process of subdivision and which passes a 0.2 mm sieve.

Test materials should not be artificially dried before testing but the presence of excessive quantities of volatile ingredients may make the test difficult to perform. To remove excessive quantities of water, preliminary drying over silica gel for 24 hours at ambient temperature is permitted. Volatile solvents should be removed according to the manufacturers recommended drying procedures. These treatments must be recorded on the data sheet at Appendix 1.

7. COMPATIBILITY TEST7.1 Preliminary test

A preliminary test is applied before testing materials such as accelerators, catalysts, amines, hardeners, alkaline materials or other reactive compounds thought to be capable of causing a rapid and hazardous reaction with the propellant under test.

Conduct the test in a fume cupboard or safety cabinet. Mix, on a watch-glass, approximately 25 mg each of the propellant and test material. Allow to stand at room temperature for one hour. If no reaction occurs, place on a boiling water bath and observe.

If the mixture darkens and fumes when cold, the test material is incompatible with the propellant and no further testing should be carried out.

If the reaction occurs when heated, the test material is likely to be incompatible with the propellant and further testing should be at the discretion of the Testing Authority.

7.2 Procedure7.2.1 Preparation of the admixture

Mix 5 ± 0.05 g of the prepared propellant with 0.5 ± 0.01 g of the test material and transfer the mixture to a glass heating tube as illustrated in Appendix 3, taking care to avoid contamination of the ground glass joint of the tube (the introduction of the propellant and the test material can be facilitated by the use of a wide-bore short stemmed glass funnel). Lightly close using a ground glass stopper and place the tube containing propellant and the test material mixture into the heating apparatus set at the specified test temperature.

Smaller sample sizes may be used, but the ratio of propellant to test material shall be maintained. The amount of sample used shall be recorded on the data sheet.

Perform each test in duplicate.

ANNEX D to
STANAG 4147
(Edition 2)

7.2.2 Preparation of the control samples.

Transfer accurately weighed samples of about 5 g, weighed to an accuracy of ± 0.05 g, of the prepared propellant to two glass heating tubes.

Lightly stopper the tubes using ground glass stoppers as illustrated in Appendix 3, and place one tube into the heating apparatus set at the specified test temperature. Store the second tube of propellant at ambient temperature. Perform each test in duplicate.

7.2.3 Sample aging.

Determine the temperature at which, after aging for 336 hours (14 days), a minimum of 20% of the propellant stabilizer is consumed but 50% depletion is not exceeded. 80°C has been found to be a suitable test temperature for most propellants stabilized with 1% or more of Ethyl Centralite or with Diphenylamine. A lower temperature is recommended for propellants stabilised with 2-Nitrodiphenylamine and for diphenylamine stabilized propellants containing nitroglycerin.

The test temperature should not exceed 80°C. If necessary a longer test time should be used to obtain the minimum of 20% stabilizer consumption.

Heat the admixture and the control propellant sample in the heating apparatus at the required temperature for 336 hours (14 days). At the end of this time period remove the samples from the heating apparatus and record any visible signs of reaction.

NOTE: The test is designed to assess the compatibility of solid materials or liquids of low volatility with solid propellants. The test can be used to assess liquid and gaseous materials and to assess the compatibility of materials with liquid nitrate ester propellants. However in both cases appropriate modifications to the test apparatus are necessary to ensure it remains leak tight for the duration of the test.

8. ANALYSIS

Determine the residual stabilizer content of the propellant samples by an appropriate analytical method such as HPLC, gas chromatography or chemical analysis. The procedures specified in STANAG 4117 have also been found suitable for the analysis of certain types of propellant containing diphenylamine, ethyl centralite or mixtures of both.

If chromatographic techniques are used, an extract of the test material shall be analyzed. Peaks due to extractable components of the test material, that may interfere with the signal from the propellant stabilizer, can then be identified.

Record the results on the data sheet (Appendix 1).

NOTE: Wherever possible the complete sample shall be analyzed for residual stabilizer content. No attempt should be made to separate the propellant from the test material. The propellant and test material are intimately mixed and obtaining a smaller, but representative sample of propellant, by subdivision of the test sample may be difficult.

NATO STANAG 4147 DATA SHEET (Side A)

(Unique Reference Number)Page ___ of ___ Page(s)

TEST SITE INFORMATION		TEST CONDITIONS	
Laboratory: (Name of Laboratory)		Test Temperature (°C):	
Date: (Date that form was completed)		Test Duration (hr):	
Test Procedure: (Name of test procedure used)		Weight of Admixture:	
Date Tested: (Date of test period)		Test Material (g):	
POC: (Point of contact)		Test Propellant (g):	
		Weight of Control Propellant:	
		Unheated (g):	
		Heated (g):	
SPECIMEN INFORMATION		SAMPLE PREPARATION	
Identification of Test Material: (Trade name and/or Identity code)		Test Material, Preparation:	
Manufacturer: (Name of Manufacturer)		Test Propellant, Preparation:	
Test Material, Specification: (state if specification controls chemical composition of product)			
Lot, Batch or Consignment Number:			
Date of Manufacture or Receipt:			
Special Storage Conditions: (If applicable)			
		TEST PROPELLANT:	
		Propellant Name:	
		Date of Manufacture: (if known)	
		Composition: (with percentages)	

Appendix 1 to
TEST 5A of
ANNEX D to
STANAG 4147
(Edition 2)

TEST 5A

NATO STANAG 4147 DATA SHEET (Side B)	
Report Reference Number: (Unique Reference Number)	Page ___ of ___ Page(s)
DIVERGENCES FROM STANDARD PROCEDURE: (if applicable include details of drying, curing or solvent removal)	
TEST RESULTS: (Record the values of A, B, C and D)	
Percentage Stabilizer in the unheated propellant sample (A%):	
Percentage Stabilizer in the propellant sample heated with the 10 % addition of test material (B%):	
Percentage Stabilizer in the heated propellant sample (C%):	
$\frac{A - B}{A - C} = D$	
DATA SENT TO: (Name and address of person receiving this information)	
COMMENTS:	
COMPATIBLE: (YES or NO)	

TEST 5A

ACCEPTANCE LIMITS FOR PROCEDURE A

The decrease in stabilizer content of the propellant in contact with the test material shall not exceed one and a half times the decrease in the stabilizer content of the propellant when heated in the absence of the test material, ie

$$\frac{A - B}{A - C} \leq 1.5$$

where:

A = stabilizer content of the unheated propellant;

B = stabilizer content of the propellant after heating in contact with the test material;

C = stabilizer content of the propellant after heating on its own.

Appendix 3 to
TEST 5A of
ANNEX D to
STANAG 4147
(Edition 2)

TEST 5A

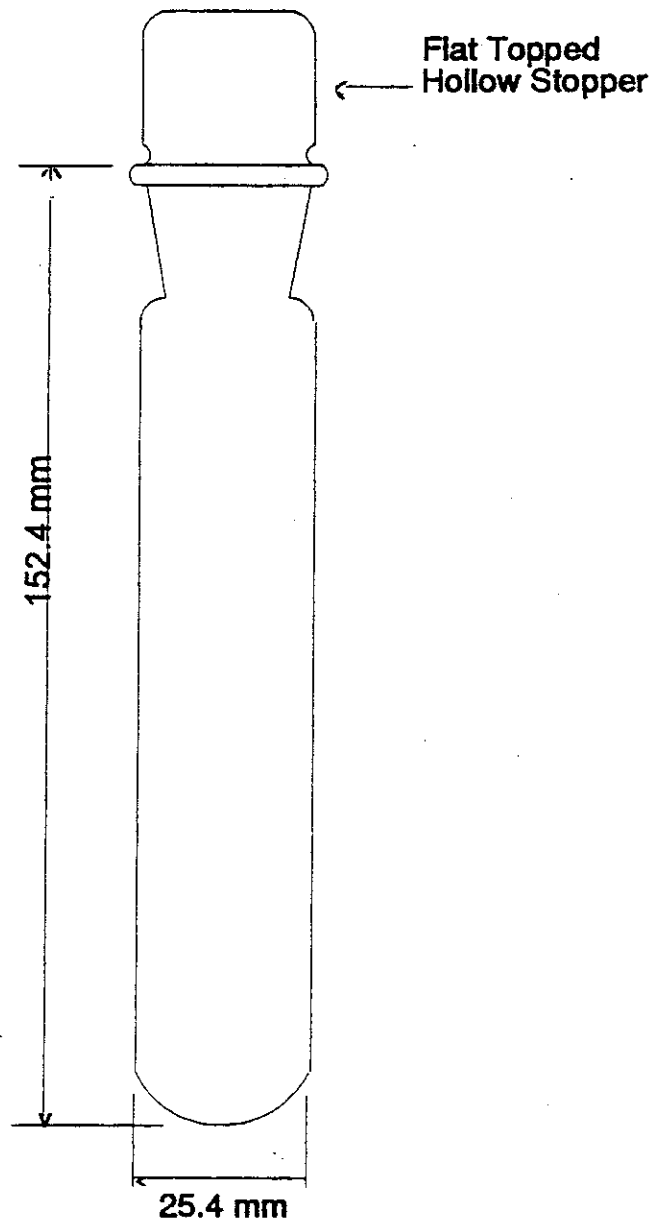


Figure 5A.1

TEST 5 - CHEMICAL ANALYSIS PROCEDURE B**ASSESSMENT OF THE COMPATIBILITY OF AMMUNITION COMPONENT MATERIALS WITH
LEAD AND SILVER AZIDE****1. SUMMARY DESCRIPTION**

Samples of the azide are heated for 672 hours (28 days) at 60°C, 95% relative humidity. Samples heated in close proximity to the material under test are compared with samples heated under identical conditions but without any test material present and samples stored at ambient temperature without the material under test.

The azide samples are analyzed to determine the level of decomposition that has occurred. The level of decomposition in all the test samples is compared and an assessment of the effect of the test material on the rate of decomposition of the azide is made.

The sample size of azide is kept as small as possible because of the sensitive nature of these explosives.

The ratio of the test material to azide is made as realistic as possible. In many applications the amount of azide present is relatively small compared to the material in question.

The test material is placed in close proximity rather than in contact with the azide as separation of the azide and test material is required before analysis can proceed and the sensitive nature of these azides would make the physical separation of a mixture too hazardous. On very rare occasions direct contact is required and different procedures for sample preparation and testing are necessary. (See Section 7 and 8 below).

WARNING.

Copper and copper containing alloys shall not be used in or tested by this test procedure, as very sensitive copper azide may be formed.

2. APPLICABILITY

The compatibility test is used for determining the reactivity of materials of ammunition components with Lead and Silver azide.

3. INTERPRETATION OF RESULTS

Materials which fail to satisfy the requirements of this test will be judged as unsuitable for use in applications where permanent direct contact with the test azide is required or where adequate precautions to guard against such contact are not taken. It shall also be judged as unsuitable for use in applications where a vapour path exists between the azide and the test material.

ANNEX D to
STANAG 4147
(Edition 2)

4. APPARATUS

- 4.1 A constant temperature oven capable of maintaining a temperature of $60 \pm 2^{\circ}\text{C}$ over a period of 672 hours (28 days).
- 4.2 A 500 cm³ Desiccator containing a saturated solution of potassium sulphate and fitted with a ceramic plate and a lid, furnished with a ground glass stopper (See Appendix 3).
- 4.3 Glass cups of about 2.4 cm in diameter x 2.7 cm deep or suitable equivalent.
- 4.4 Glass vessel of about 7.5 cm in diameter x 4.3 cm deep or suitable equivalent.
- 4.5 Compatible petroleum jelly or silicone grease.
- 4.6 Automatic potentiometric titrator. (see Appendix 4 for further details)

5. PREPARATION OF AZIDE TEST SAMPLE

Azide samples shall be used without further preparation.

6. PREPARATION OF MATERIAL TEST SAMPLE

The material used for the test shall be, as far as is practicable, in the condition proposed for use with the azide. Where the intended use requires the test material to be applied in an intermediate (uncured or partly cured) condition, the compatibility test shall be conducted using the test material in this intermediate state.

Most solid materials require to be subdivided for testing. Wherever possible, break, rasp or cut solid material into small pieces so that they can be readily packed into the reaction vessel and present as large a surface area as possible.

If direct contact between the test material and the azide is required two small discs, the same diameter as the glass cup used to contain the azide, are prepared for each test. This procedure is only possible for materials that can be produced as thin film. eg sheet polymer or thin films of paint.

Test materials should not be artificially dried before testing but the presence of excessive quantities of volatile ingredients may make the test difficult to perform. Volatile solvents should be removed according to the manufacturers recommended drying procedures. These treatments must be recorded on the data sheet at Appendix 1.

7. COMPATIBILITY TEST

Place accurately weighed samples of about 50 mg of the azide, weighed to an accuracy of ± 0.1 mg into four small glass cups.

Place two of the filled glass cups on a ceramic plate in a 500 cm³ desiccator (see Appendix 3).

Place the remaining filled glass cups in a larger glass vessel and distribute the sample of test material carefully around them. Unless otherwise specified a 10 g sample of test material is normally used. However the nature of the material or the component design may necessitate

the use of alternative sample sizes. The amount of sample used shall be recorded on the data sheet at Appendix 1.

Place the large glass vessel on a ceramic plate in a second 500 cm³ desiccator (see Appendix 3).

NOTE: If direct contact between the test material and the azide is required, a prepared disc of material is placed in the bottom of each of two small glass cups. An accurately weighed sample of azide is carefully placed on each disc and a second disc of material placed on top. The small glass cups are then placed on a ceramic plate in a 500 cm³ desiccator (see Appendix 3).

Maintain the atmosphere inside the desiccators at 95% relative humidity by placing a small quantity of saturated potassium sulphate solution, containing some undissolved crystals, in the bottom.

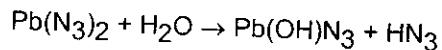
Place the desiccators, with the stoppers removed, in a temperature controlled oven set at 60 ± 0.2°C. Allow the desiccators and contents to reach the temperature of the oven (approx. 4 hours) then replace the glass stoppers and heat for 672 hours (28 days). At the end of this time period remove the samples from the oven and record any visible signs of reaction. Reserve samples of the azide and store at ambient temperature and humidity for the duration of the test.

All deviations from the standard test procedure shall be recorded on the test data sheet (Appendix 1).

8. ANALYSIS

8.1 Introduction

During aging Lead and Silver azide can be hydrolysed to produce hydrazoic acid, with the resultant reduction of active azide. Under normal conditions an equilibrium is set up.

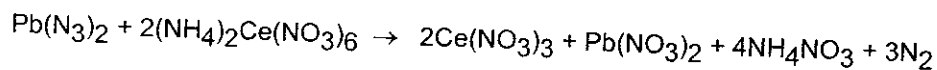


An incompatible material can alter the balance of this reaction either by the introduction of moisture, or other products, such as acidic products, or by reaction with and resultant removal of hydrazoic acid. Both reactions can accelerate the loss of azide.

This method measures the percentage azide present, after aging under the conditions described in Section 7 above. The percentage azide remaining in the samples aged with the material under test, is compared with the percentage azide in the samples aged under identical conditions but without the presence of the material under test.

8.2 Summary description

A titrimetric method making use of the following reaction is used. (Shown for lead azide).



An automatic micro titrator using a redox electrode is employed.

ANNEX D to
STANAG 4147
(Edition 2)

A known excess of standard Cerium IV ammonium nitrate solution is added to an azide slurry or solution and allowed to react. The excess Cerium IV ammonium nitrate is back titrated with standard Iron II ammonium sulphate.

8.3 Preparation of standard solutions

Standard solutions of Cerium IV ammonium nitrate and Iron II ammonium sulphate are prepared and standardized as follows:

8.3.1 Reagents

- Solution (0.025 M) of ortho-Phenanthroline-Iron II ammonium sulphate indicator, prepared by dissolving 1.487 g of ortho-phenanthroline monohydrate in 100 cm³ of an aqueous solution of Iron II ammonium sulphate, containing 0.980 g of Iron II ammonium sulphate hexahydrate per 100 cm³.
- Dilute perchloric acid solution, prepared from 60 cm³ of 70-72% perchloric acid solution diluted to 1 litre with distilled water.
- Standard Sodium oxalate, analytical grade.

8.3.2 Cerium IV ammonium nitrate solution (approximately 0.1M)

Dilute 60 cm³ of 70-72% perchloric acid solution to approximately 700 cm³ with distilled water and add this solution to 60 g of analytical reagent grade (99 %) Cerium IV ammonium nitrate. Stir to dissolve and make up to 1 litre with water, mixing thoroughly.

8.3.3 Standardization of Cerium IV ammonium nitrate

Transfer a weighed portion of 0.25-0.30 g of standard sodium oxalate to a 250 cm³ beaker. Add 100 cm³ dilute perchloric acid solution. Stir the mixture until the salt has completely dissolved. Cool the solution to room temperature, add 2 drops of the ortho-phenanthroline-Iron II ammonium sulphate indicator and titrate the solution with Cerium IV ammonium nitrate solution until the colour changes from a faint red to faint blue.

The molarity of the Cerium IV ammonium nitrate solution is given by:

$$\frac{AW}{6.701B}$$

Where:

A	=	purity in percent of the sodium oxalate;
W	=	weight of sodium oxalate taken (g)
B	=	amount of Cerium IV ammonium nitrate solution used (cm ³)

8.3.4 Iron II ammonium sulphate solution (0.05M)

Dilute 75 cm³ 70-72% perchloric acid with 750 cm³ of distilled water. Add 19.6 g of Iron II ammonium sulphate hexahydrate (FeSO₄(NH₄)₂SO₄·6H₂O) and mix. Make up to 1 litre with distilled water.

The Iron II ammonium sulphate solution is stored over a zinc/ mercury amalgam.

8.3.5 Standardization of Iron II ammonium sulphate

Measure approximately 40 cm³ of standard Cerium IV ammonium nitrate solution into a 250 cm³ beaker and dilute to 100 cm³ with the dilute perchloric acid solution. Add two drops of ortho-phenanthroline-Iron II ammonium sulphate indicator and titrate the solution with the Iron II ammonium sulphate solution until a colour change from light blue to red is observed.

The volume of standard Cerium IV ammonium nitrate solution equivalent to 1 cm³ of Iron II ammonium sulphate solution is found by dividing the volume of standard Cerium IV ammonium nitrate solution taken by the volume of Iron II ammonium sulphate used in the titration.

8.4 Procedure for determining the percentage azide present

The aged azide sample is very carefully transferred to a tared titration vessel and accurately weighed to ± 0.01 mg. 35 ± 0.1 cm³ of 1M perchloric acid is added.

NOTE: To limit the production of HN₃ it may be necessary to maintain the temperature of the solution below 15°C, or use a closed system.

The titration vessel is rapidly attached to the titrator. 5 ± 0.1 cm³ of 0.1 M Cerium IV ammonium nitrate is immediately and accurately dispensed from the burette. The mixture is stirred constantly for three minutes. All the azide present reacts leaving a small excess of Cerium IV ammonium nitrate. The excess Cerium IV ammonium nitrate is back titrated with 0.05 M Iron II ammonium sulphate and the end point recorded. The percentage azide present is given by :

$$\frac{K(A-BC)M}{20W} = \% \text{ Lead Azide}$$

$$\frac{K(A-BC)M}{10W} = \% \text{ Silver Azide}$$

Where:

- A = cm³ of Cerium IV ammonium nitrate solution taken.
- B = cm³ of Iron II ammonium sulphate solution required.
- C = cm³ of Cerium IV ammonium nitrate solution equivalent to 1 cm³ of Iron II ammonium sulphate solution.
- M = molarity of the Cerium IV ammonium nitrate solution.
- W = weight of azide transferred to the titration vessel (mg).
- K = molecular Weight of the azide.

Record all the results on the data sheet at Appendix 1.

NATO STANAG 4147 DATA SHEET (Side A)

Page ___ of ___ Page(s)

<div style="float: right; text-align: right;"> Page or Page(s) </div> TEST SITE INFORMATION		TEST CONDITIONS	
Laboratory: (Name of Laboratory)	Test Temperature (°C):		
Date: (Date that form was completed)	Test Time (days):		
Test Procedure: (Name of test procedure used)	Weight of Admixture:		
Date Tested: (Date of test period)	Test Material (g):		
POC: (Point of contact)	Test Azide (g):		
	Weight of Control Azide:		
	Unheated (g):		
	Heated (g):		
SPECIMEN INFORMATION		SAMPLE PREPARATION	
Identification of Test Material: (Trade name and/or Identity code)	Test Material, Preparation:		
Manufacturer: (Name of Manufacturer)			
Test Material, Specification: (state if specification controls chemical composition of product)			
Lot, Batch or Consignment Number:			
Date of Manufacture or Receipt:			
Special Storage Conditions: (if applicable)			
	TEST AZIDE		
	Azide Name:		
	Date of Manufacture: (if known)		
	Composition: (with percentages)		

TEST 5B

NATO STANAG 4147 DATA SHEET (Side B)	
Report Reference Number: (Unique Reference Number)	Page ___ of ___ Page(s)
DIVERGENCES FROM STANDARD PROCEDURE: (if applicable include details of drying, curing or solvent removal)	
TEST RESULTS: (Record the values of E, F, G and D)	
Percentage Azide in the unheated azide samples (E%):	
Percentage Azide in the heated samples (F%):	
Percentage Azide in the samples heated with 10 g of test material (G%):	
$D = F - G$	
DATA SENT TO: (Name and address of person receiving this information)	
COMMENTS:	
COMPATIBLE: (YES or NO)	

Appendix 1 to
TEST 5B of
ANNEX D to
STANAG 4147
(Edition 2)

TEST 5 B

ACCEPTANCE LIMITS FOR PROCEDURE B

The difference in the percentage azide (D) in the samples heated with 10 g of test material and heated alone (F - G) shall not be greater than 3% for lead azide and 2% for silver azide where:

- F = percentage azide in a 50 mg sample of azide heated for 672 hours (28 days) at 60°C;
- G = percentage azide in a 50 mg sample of azide heated for 672 hours (28 days) at 60°C in the presence of a 10 g sample of the test material.

TEST 5B

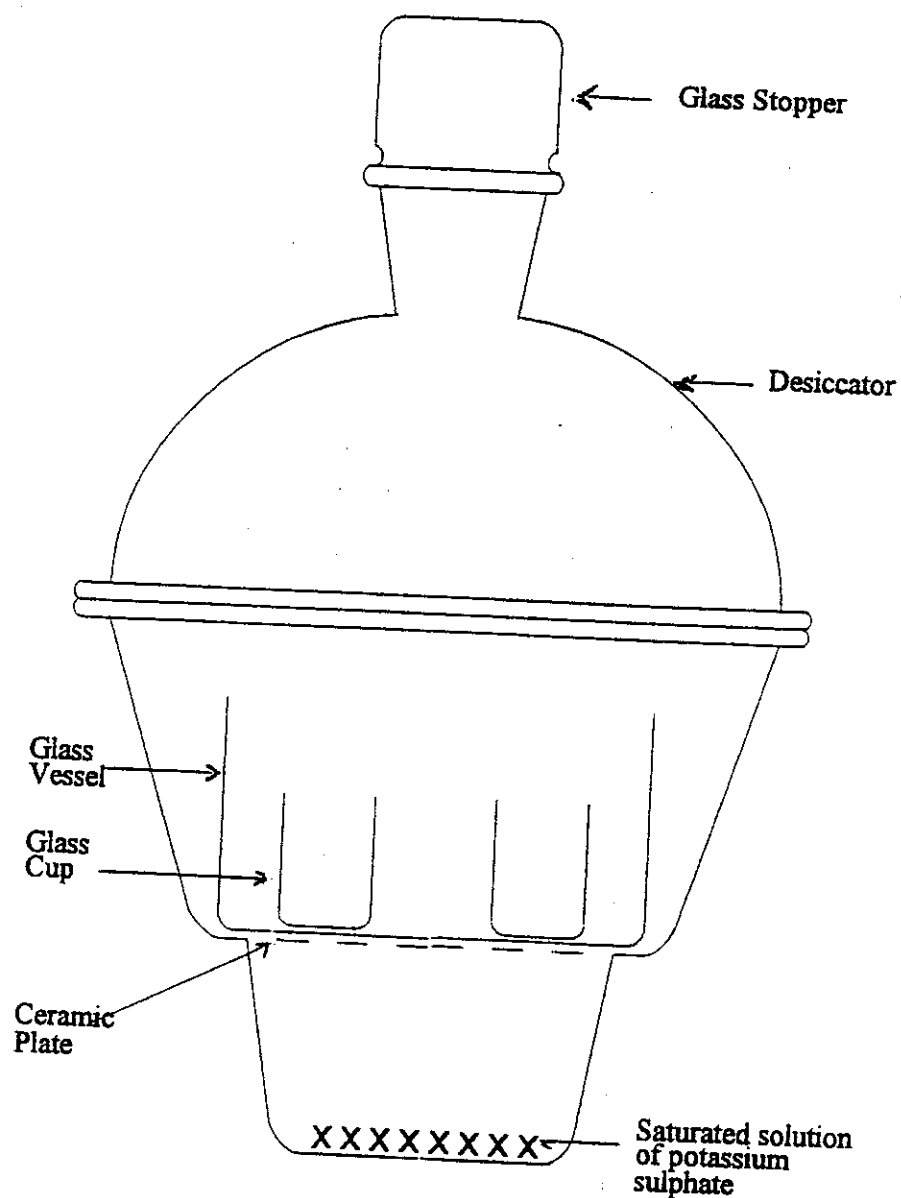


Figure 5B.1

Appendix 1 to
TEST 5B of
ANNEX D to
STANAG 4147
(Edition 2)

TEST 5B
SOME GUIDANCE NOTES ON THE USE OF AN AUTOMATIC TITRATOR
FOR THIS PROCEDURE

1. Any standard automatic potentiometric titrator can be used. The following notes are based on the use of a Mettler DL40 titrator for the procedure described in test 5B.
2. Once the azide has been transferred to the titration vessel and weighed, the first reagent to be added is 1M perchloric acid. This is dispensed from a reservoir using an auxiliary pump. The pump is set at a constant speed and is controlled by the titrator. The speed of introduction should be rapid, but slow enough to prevent splashing. Once a suitable speed of addition has been established, the parameters should be recorded and used at all times.
3. The Mettler DL40 has a standard stirring time after addition of the first reagent that cannot be overridden. The time is therefore set at the minimum value, which is 1 second.
4. The second reagent to be added is 0.1M Cerium IV ammonium nitrate. This is dispensed automatically from one of the auto titrator's burettes at a constant speed, dictated by the titrator.
5. The azide/Cerium IV ammonium nitrate solution is stirred for three minutes to allow complete reaction. This time must be the same for each sample analyzed. Experience has shown that three minutes is the minimum amount of time required for total reaction to occur. The stirrer speed is controlled by the titrator. A stirrer speed just slow enough to avoid creating a vortex in the solution should be aimed for. Using a Mettler DL40 a speed set to 50% of maximum has been found to be suitable.
6. The third reagent to be added is 0.05M Iron II ammonium sulphate. This is dispensed using one of the titrator's burettes. By this stage all the azide has reacted and timing is no longer critical.

COMMENT 1: The addition of Cerium IV ammonium nitrate directly to the solid azide is not advisable. The Cerium IV ammonium nitrate starts reacting instantly with the azide and experience has shown that spitting can occur. The reaction is more controlled if 1M perchloric acid is mixed with the azide first. Under these conditions the effervescence produced when the Cerium IV ammonium nitrate is added is more controlled and there is no spitting.

COMMENT 2: Perchloric acid solutions can themselves react with Lead and Silver azides with the production of hydrazoic acid (HN_3), but experience has shown the reaction to be relatively slow, under the conditions used in the titration procedure described.

The compatibility test described in Test 5B is a comparative test. Therefore, to ensure this side reaction has no effect on the overall results of the compatibility test it is imperative that the timing of the titration procedure, to the addition of the Iron II ammonium sulphate solution, is controlled and is the same for all the azide samples analyzed. Any reaction between the azide and perchloric acid in each sample is the same and is eliminated when determining the value of D at Appendix 2.

RATIFICATION AND IMPLEMENTATION DETAILS
STADE DE RATIFICATION ET DE MISE EN APPLICATION

N A T I O N A L P A Y S	NATIONAL RATIFICATION REFERENCE	NATIONAL IMPLEMENTING DOCUMENT	IMPLEMENTATION/MISE EN APPLICATION					
	REFERENCE DE LA RATIFICATION NATIONALE	DOCUMENT NATIONAL DE MISE EN APPLICATION	INTENDED DATE OF IMPLEMENTATION			DATE IMPLEMENTATION WAS ACHIEVED		
			DATE ENVISAGEE DE MISE EN APPLICATION			DATE EFFECTIVE DE MISE EN APPLICATION		
			NAVY MER	ARMY TERRE	AIR	NAVY MER	ARMY TERRE	AIR
BE								
CA	2441-4147 (DAPM 4-4) of/du 18.10.00	STANAG	12.00	12.00	12.00			
CZ	6/2-18/2000-1419 of/du 26.07.00	STANAG		12.01	12.01			
DA*	FKO MAM3 204.69-S4147 0004926-003 of/du 05.07.00	STANAG	09.01	09.01	09.01			
FR*	DGA/INSP Nr.030823 of/du 07.02.01	AFNOR NF T 70-516 & NF T 70-517, & CMP-320 & CMP-321	03.01	03.01	03.01			
GE	BMVg - Fü S IV 1 - Az 03-05- 60 of/du 19.04.01	STANAG	10.01	10.01	10.01			
GR	Ø 049.Ba/410434/Σ381 of/du 17.04.00	STANAG	01.01	01.01	01.01			
HU								
IT								
LU	BO 473/00 of/du 29.03.00	Not implementing / Ne met pas en application						
NL*	M2001001971 of/du 25.04.01	STANAG				09.00	06.01	06.01
NO								
PL								
PO								
SP	OMD Nr. 323/01568/2001 of/du 17.01.01	STANAG	06.01	06.01	06.01			
TU								
UK	D/Dstan/12/15/4147 of/du 28.04.00	STANAG	11.01	11.01	11.01			
US ⁺	OUSD(A&T) of/du 16.11.00	MIL-STD-1751	11.00	11.00	11.00	11.00	11.00	11.00

* See overleaf reservations(*)/comments (+)
Voir au verso réserves (*)/commentaires (+)

STANAG 4147
(Edition 2)

RESERVES/RESERVATIONS

- FRANCE Annex D, test 1, procedure A, para 8.1 and Annex D, test 1, procedure B, para 8.1:
- For safety reasons, the mass of the samples to be tested is limited to 1 g, in compliance with the procedures described in the applicable national documents and AFNOR standards NF T 70-516 and NF T 70-517
- FRANCE Annexe D, test 1, procédure A, paragraphe 8.1 et annexe D, test 1, procédure B, paragraphe 8.1:
- Pour des raisons de sécurité, la masse des échantillons pour essai est limité à 1g, conformément aux procédures décrites dans les documents nationaux d'application, normes AFNOR NF T 70-516 et NF T 70-517.*
- THE NETHERLANDS Data developed in accordance with this Stanag shall be made available by the Netherlands to other NATO nations, participating in a collaborative weapon development or procurement programme upon receipt of a request submitted through appropriate national channels.
- PAYS-BAS *Les données obtenues conformément au présent STANAG pourront être communiquées par les Pays-Bas aux autres pays participant au développement ou au programme d'acquisition d'une arme en collaboration, sur demande soumise par les voies nationales appropriées.*

=====

COMMENTS/COMMENTAIRES

- DENMARK STANAG will be used as the implementing document.
Danish authorities consider this STANAG releasable to PfP nations, other nations and organizations.
- DANEMARK *Ce STANAG sera utilisé comme document de mise en application.
Les autorités danoises estiment que ce STANAG peut être communiqué aux pays du PPP, à d'autres pays et à d'autres organisations.*